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RADIOCARBON CHRONOLOGY OF THE ANCIENT SETTLEMENT IN THE GOLAN HEIGHTS AREA, ISRAEL

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ABSTRACT. Carbonate binders from mortars and plasters as well as charcoal fragments sampled at the ancient settlement of Hippos (Sussita) have been subjected to radiocarbon dating by gas proportional counting (GPC) and accelerator mass spectrometry (AMS). Hippos is situated on the east coast of the Sea of Galilee (32°46′N, 35°39′E) at the top of a hill in the Golan Heights area, Israel. According to historical-archaeological data, the town had functioned since the 3rd century BC until AD 749, when it eventually crumbled into ruins after an earthquake. The appropriate sample selection and preparation based on the results of petrographic observations permitted us to distinguish different phases involved in the expansion of the settlement. More than 200 samples were taken from the settlement and subjected to petrographic and chemical analyses. Of the 200 total samples, about 20 were selected for dating. Here, we present the first 10 results of ^{14}C dating carried out for Hippos. The oldest sample dated thus far gave an age corresponding with the 2nd century BC to 1st century AD—probably indicating an old Roman temple, on the base of which the North-West church (NWC) was later erected. The next dates extend up to the 8th century AD, the age related to the last phase of settlement inhabitation. Research is continuing as new excavations take place.

INTRODUCTION

This paper presents the results of radiocarbon dating at the site of Hippos (Sussita), Israel. The previous chronology of the settlement was based on artifactual evidence (ornaments, trims, inscriptions, and coins characteristic for this period) and on information from historical sources (Flavius 1993). The study is interdisciplinary and the cooperation of archaeologists, geologists, and physicists coupled with the interpretation of the literature allows for determination of the phases of the settlement development. The research simultaneously addresses the general problem with dating mortars and plasters. The difficulties in ^{14}C dating mortars are connected with the presence of "dead carbon" and the success with which it can be excluded from consideration (Baxter and Walton 1970; Folk and Valastro 1976, 1979; Van Strydonck et al. 1986; Heinemeier et al. 1997; Sonninen and Jungner 2001; Hodgins et al. 2006). From Hippos, where the excavations are continuing, the carbonate binders and charcoal separated from mortars and plasters have been dated. The peculiar relationship between the mortars and the time of building construction makes them very desirable dating material for archaeologists. When mortars are inaccessible, plasters are used for dating, although they are not directly connected with the time of construction. Obviously, taking into account the possibility of subsequent repairs to the building, material selection before dating must be very careful.

Proper selection of samples with preparation adjusted to the composition, together with mechanical and chemical methods of separation, help eliminate the known error sources and enable the dating of mortars. Dating of the mortars and plasters from Hippos had been preceded by a detailed analysis of their composition, so as to exclude the possibility of age overestimation due to the presence of the old carbonate aggregate or underestimation of the effect of secondary recrystallization of the binder. The chosen samples (Hip10, Hip61) have been dated twice, via accelerator mass spectrometry (AMS) and gas proportional counting (GPC), each time using slightly different preparation methods connected with the requirements of the particular counting technique. We aimed to verify not only the results but also the applied preparation and sample selection methods.

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Previous archaeological suppositions concerning different phases of the settlement development were based on fragments of the walls gradually uncovered during the excavations. The investigations in Hippos were initiated at the end of the 19th century by a German traveler, Gottlieb Schumacher. Excavations were begun by the Israeli Department of Antiquities between 1950–55. After a long pause in excavations, research on the system of water supply was conducted by an Israeli-German group in 1990. In 2000, systematic excavations began within the confines of the project led by Prof Arthur Segal from the University of Haifa, Israel.

SITE DESCRIPTION AND SAMPLING

The ancient settlement (in Greek named "Hippos," in Aramaic "Sussita") is situated at the top of the hill about 350 m above the water level of the Sea of Galilee (Figure 1). Hippos was founded in the 3rd century BC. Under Roman reign, it belonged to Dekapolis, and in Byzantine times it prospered as the seat of a bishopric (Segal et al. 2004). Hippos is mentioned in historical sources, e.g. by Josephus Flavius and Plinius the Older. The town collapsed into ruin following an earthquake in AD 749. The time of the settlement destruction was determined by a combination of historical and archaeological information combined with geological data (Amiran et al. 1994; Marco et al. 2003; Ben-Avraham et al. 2005). Hippos is located in the zone of the Dead Sea transform—a left-lateral fault running between the Arabia and the Sinai tectonic plates. The Dead Sea transform links the Red Sea ocean floor spreading zone with the continental collision zone of the Taurus and Zagros mountains in SE Turkey (see Figure 2; Ellenblum et al. 1998; Niemi et al. 2001; Marco et al. 2003). This still-active fault zone is associated with earthquakes; thus, it contributes significantly to the history of population and the architecture of the region. The Dead Sea region provides a rich paleoseismic record and the earthquakes left many traces in the ancient architecture (Ellenblum et al. 1998): for example, the earthquake and fires in 31 BC caused the depopulation of Qumran; the traces of the earthquakes are evident also for Hippos (AD 749), Ateret (1202 and 1759), and Jericho (1927). The signs of devastation (in the form of destroyed buildings, overturned tiers of columns, fractured walls) left by the AD 749 earthquake are found in numerous settlements situated along the fault zone, e.g. at Jericho, Kursi, Tiberias, Hippos, and many others (Flavius 1993; Amiran et al. 1994; Guidoboni 1994; Marco et al. 2003). Historical literature about the earthquakes, and their evidence in the architecture and seismic data, coupled with the knowledge about active fault zones, enable us in many cases to determine precisely the age of an earthquake and, by the same means, the destruction induced by that event. These data indicate a 100-km-long rupture segment spreading between the Kinneret and the Dead Sea pull-apart basin. It can generate earthquakes of magnitude >7 on the Richter scale. In Hippos itself, the magnitude was estimated as above 9, that is, "causing enormous devastations" (Russell 1985; Guidoboni 1994; Marco et al. 2003). On the basis of all the available sources of information, the last phase of occupation at Hippos was determined to be the 8th century AD.

The geological setting of the Golan Heights, and indeed the wider region, is distinctly reflected in the building materials used in the settlement, with walls made of basalt and limestone blocks. As a binding material, mainly carbonate mortars had been used and they were sampled for ^{14}C dating. The aim of the ^{14}C dating has been to verify the estimations made by archaeologists and to indicate the stages of development and extension of the settlement.

Until now, the divisions of the extension phases in particular buildings were based on the wall stratigraphy, decorative motifs of mosaic floors, and discovered artifacts. For example, the excavations conducted in 1950–55 by the Israeli Department of Antiquities revealed part of a church (the Hippos cathedral) together with a baptisterion, dated to AD 591 by the inscription on the floor (Epstein and

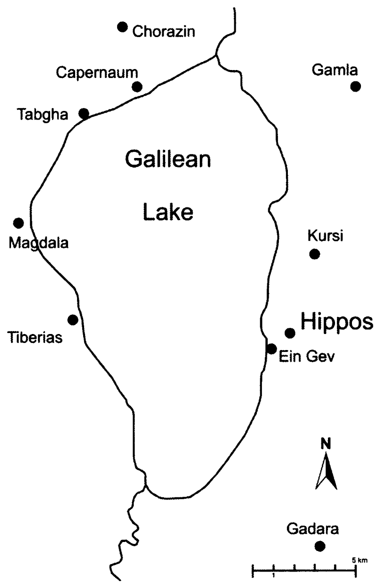

Figure 1 Location of Hippos

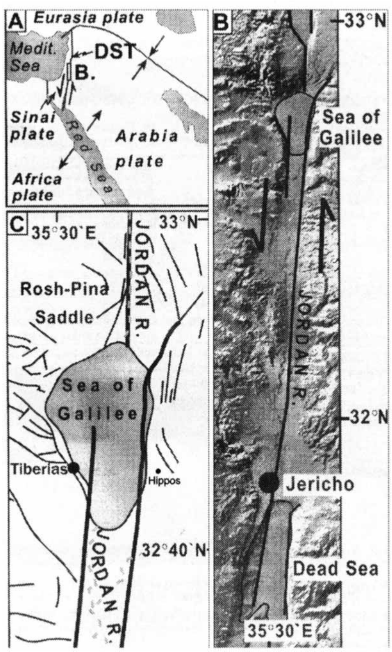

Figure 2 Fault zone in research area: A) Middle East tectonic plates; B) Dead Sea transform fault (DST) between the Sea of Galilee and the Dead Sea (shaded relief from Hall [1994], in Marco et al. 2003); C) faults around the Sea of Galilee (according to Marco et al. 2003).

Tzaferis 1991). The subsequent archaeological work showed that one of the churches exposed during the excavations was built on the former Roman temple site. This 3-aisle church is located in the northwest sector of Hippos, parallel to the main street (Figure 3). During several seasons of excavations, on the basis of the wall stratigraphy, ceramics, and artifacts, archaeologists distinguished 3 chronological phases of construction and development of the church (Młynarczyk 2001; Segal et al. 2004). The first phase was estimated as the 5th century AD. The second phase was determined by archaeologists as the last quarter of the 6th century, on the basis of the repertoire of decorative motifs occurring on the mosaic floors made in those times. The third phase was connected with the extension of the chancel due to the installation of balustrades in the side aisles, most probably during the 7th century AD. The church was open until the earthquake in AD 749 (Młynarczyk 2000, 2001). In the Byzantine period, the settlement became a significant Christian center and at least 3 churches were constructed. The mono-apsidal North-East Church (hereafter NEC) is located between the cathedral with its tri-apsidal baptistery and the North-West Church (hereafter NWC).

Tracing the history of the settlement on the basis of the literature and the conducted excavation works, the area of the NWC was selected as the first site for the datings. Many factors were taken into account—for example, the notable time range of the excavated walls, which is representative for the whole settlement. From among all the samples, the group representing possibly different stages of the construction development has been chosen. In order to compare the functioning time of the NWC and the nearby NEC, a sample from the latter has also been dated. Samples were taken at the following sites (see Table 1 and Figure 3): collection pool in the agricultural installation to the south of the diakonikon (samples Hip2, Hip2/ch); southern aisle, by the balustrade, northern face (samples Hip10, 10H); "floor pour" (a layer of the floor made to level uneven concrete floors) of a channel exposed at the chancel area, loc. 257 (sample Hip14); façade of the pastophorium northern wall (samples 61Hip, 61H); and the passage between the main and northern aisle, pier at the western wall (sample 70H), all from the NWC site and its vicinity and the plaster from the apse, NEC (sample A1). The dated material consisted mainly of carbonate binders from mortars and plasters. Samples 2, 14, and 61 are building mortars, while samples 10, 70, and A1 are finishing plasters.

Figure 3 Aerial photograph, viewed from the north, toward the center of Hippos (photo by A Greizer, from Segal et al. 2004), with the sampling sites marked by the authors: North-West Church (NWC) and adjoining objects in Hippos with location of dated samples.

MATERIALS AND METHODS

The carbonate binder from mortars and plasters as well as the charcoal fragments sampled at the settlement have been analyzed in the conventional ^{14}C laboratory the Gliwice Radiocarbon Laboratory and in the Poznań AMS Radiocarbon Laboratory.

Building mortar is a mixture of binder, aggregate, and water, possibly containing the coloring and sealing admixtures, and is used in construction work. Grains of the aggregate form the matrix of the mortar, hindering its shrinkage and cracking; cement grout, which develops after adding water to cement, coats the sand and gravel grains and fills the gaps between them. Today, as in the past, the choice of the proper mortar and the right proportion of its ingredients is undertaken while considering the characteristics of the products, their destination, and future operational conditions. Thus, mortars are the material applied for connecting elements of the building, formed and used during the building construction (they bond the elements of the walls).

On the other hand, plasters are layers coating the surfaces of walls, ceilings, and columns inside and outside buildings. Therefore, in the context of the datings, the difference between plasters and mortars is important because of (when using appropriate sample selection) the direct correlation of mortar age with the time of the building erection; whereas in the case of plaster one should treat with caution the interpretation of the obtained ages. Obviously, during the period of settlement inhabitation both those building materials may be subjected to repairs; however, much more frequently repairs occur to plasters due to their finishing function.

Under certain conditions, the dating of carbonate binders makes possible the determination of the true age of a certain sample. ^{14}C dating of lime mortars and plasters is based on determining the present ^{14}C concentration of CO_2 by mortar carbonates in the hardening process (Folk and Valastro 1979; Pachiaudi et al. 1986; Van Strydonck et al. 1986; Sonninen and Jungner 2001; Hale et al. 2003; Tripp et al. 2004; Nawrocka et al. 2005). However, to obtain satisfactory results it is necessary to obtain precise petrographic observations in order to eliminate carbonate aggregates. The presence of carbonate aggregates, as well as fragments of limestones that are not completely burnt, may be a cause of considerable overestimation of the age. The dating of mortars from the Dead Sea area is a good case in point. In this case, the presence of carbonate aggregate, despite the efforts to separate it (by freezing, warming, and separating under stereomicroscope) and despite applying a reservoir age correction, the ages determined for the entire series (without aggregate separation) were much too old (Nawrocka et al. 2005). A significant element of sample selection is also the analysis and observation of the samples from the point of view of possible secondary recrystallization of binder, the process which can "rejuvenate" the ages obtained by dating. Thus, petrographic observations are an important element of sample selection before dating, since they enable us to choose the appropriate methods of preparation suitable for any given mortar. After recognition of the composition and size of the mortar and plaster ingredients, an attempt to separate different fractions can be made. The separation of fractions from mortars for dating seems to be promising (Van Strydonck et al. 1986; Heinemeier et al. 1997; Sonninen and Jungner 2001). The method of fractional separation of the mortars takes into account the relationship of speed of reaction versus grain size and is based on the difference of the reaction rates between old carbonate aggregate (limestone, dolomite) and also carbonate mortar binder. Usually, limestone fragments react slower than the mortar binder (Jędrzejewska 1960; Van Strydonck et al. 1986; Sonninen and Jungner 2001). The choice of fractions in every case should be made individually depending on the mortar composition, type, and size of the applied aggregate. In the case of the samples from Hippos selected for dating, the aggregate size in the analyzed samples ranged from 0.2 to 1.5 mm in diameter. In this study, the technique proposed by Sonninen and Jungner (2001) has not been applied in all cases. The reason is that for some particular fractions, the amount of the material remaining was not sufficient after sieving. This did not cause problems for the ages determined, because the main part of the carbonate aggregate that could cause overestimation had been removed by sieving out the coarse aggregate. The final separation was controlled under stereomicroscope.

The fractions 80–100 μm for sample 61H (Gd-18388) and 45–100 μm for sample A1 (Gd-17381) have been dated. In those samples, the carbonate aggregate is composed mainly of different limestones. In addition, the aggregate contains crushed fragments of basaltic rocks, flints, and sandstones. Sample 61 is a white lime mortar containing coarse-grained, basaltic-carbonate aggregate dominated by different limestone types: massive pelitic limestones, crystalline limestones, and oncolitic limestones. Sample Hip2 contained a large amount of fine basaltic aggregate with a small portion of limestone aggregate. The carbonate material of sample Hip2, destined for dating by AMS (Poz-5016), was not separated into fractions. The sample was delicately crushed, then the single mortar fragment was separated using the stereomicroscope. The fragment was dissolved, and the gas was collected only in the first 5 s of the reaction. The AMS dating of sample 61Hip (Poz-16078) was carried out in a similar way. The attempt to date such prepared fragments aimed to verify the possibility of dating a sample with a known composition and size of aggregate without fraction separation based on the principle that more sensitive binder dissolves faster than harder limestone aggregate (Van Strydonck et al. 1986; Sonninen and Jungner 2001). Therefore, the gas collection for dating during the first seconds of the reaction was of interest. Additionally, for sample Hip2/ch the measurement for charcoal has been performed by the same technique. For sample 61, the separated fraction 80–100 μm was dated using GPC, Gd-18388 (charcoal was absent in the sample). For sample Hip2 (Poz-5016), the gas was collected during the first 5 s of the reaction with orthophosphoric acid, whereas for 61H (Poz-16078) the time was lengthened to 15 s due to the insufficient amount of the gas from the first 5 s. Figure 4 displays the fragment of the mortar from the ruins of Hippos in microscopic view, and presents the main components of the aggregate. The samples selected for dating are the carbonate mortars and plasters with a diverse percentage composition of aggregate and binder.

The samples were taken with strict respect to the wall stratigraphy and occurring lithological changes. During thin section observations, we checked if there was compositional diversity within a single wall or other traces of reconstruction or renovation. The samples were also carefully analyzed regarding possible secondary recrystallization, which could cause “rejuvenation” of the ages obtained by dating. The samples finally selected for dating were devoid of these inconveniences. The next condition necessary to obtain the construction age from the mortars is a relatively short time of the hardening process. Therefore, the sampling was conducted with special consideration to the depth in the wall. The outer layer, if present, was removed from the samples.

The samples can be divided into 2 main groups. The first group consists of lime mortars (samples 2, 14, 61) and plasters (A1) being a mixture of binder and aggregate, including the carbonate-basalt aggregate and carbonate binder in different proportions. The second group is constituted by almost pure lime plasters, containing only the empty spaces after straw (samples 10, 70). For the first group, the preliminary preparation, conducted after determining petrographic structure, type, and size of the aggregate, was executed. Preparation consisted of sieving the samples and dividing them into the fractions with specific granulation. Fractional separation of the remaining samples was not necessary due to the lack of carbonate aggregate in their composition. For the purpose of comparing the results, some samples (samples 10, 61) have been dated both by GPC in Gliwice (Gd-12823) and AMS in Poznań (Poz-7417). Conduction of the measurements for the samples from the interior of the NWC (sample 10) and the adjoining NEC (sample A1)—both represented finishing plasters—also enabled us to verify if they could be coeval. The remaining samples represent different fragments of the walls, mainly in the NWC and its surroundings. They indicate diverse phases of the building development.

The selection of samples, mineralogically diversified and coming from the different phases of the construction, allows us to verify the estimation made by archaeologists, based on stratigraphy of the walls, and to verify the possibility of application of the method for dating mortars with diverse petrographic composition and originating in different time ranges (what is connected with slightly different technology). In AMS, the mortar samples were diluted (by the reaction with phosphoric acid

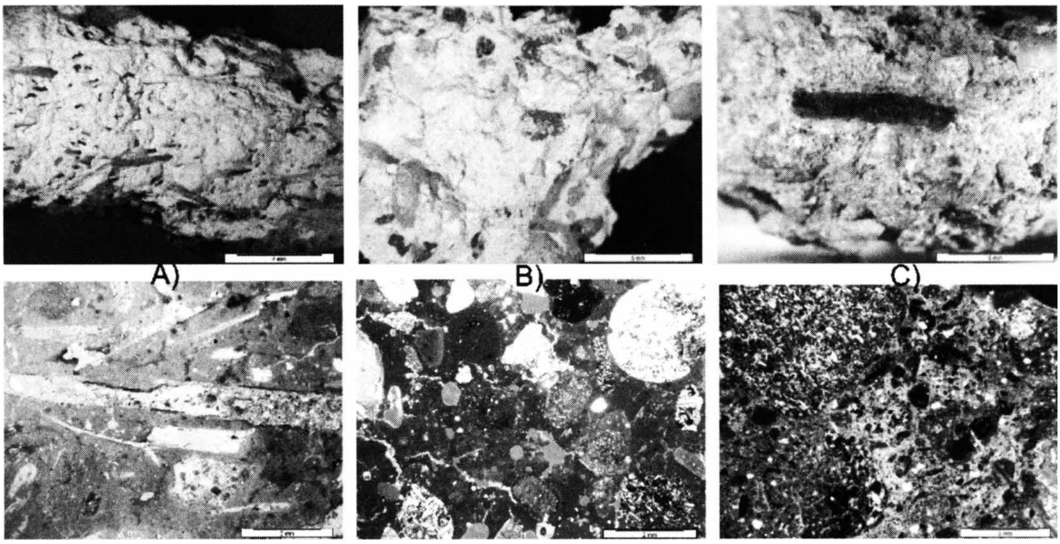

Figure 4 Micro- and macroscopic views of the selected mortars, representing 2 main types: A) "pure" carbonate lime plasters; B) and C) mortars containing mainly basalt-carbonate aggregate; C) sample Hip14, from which charcoal was dated. Respectively, the micro- and macrophotographs are A) photo of sample Hip10, B) photo of sample 61H, and C) photo of sample Hip14.

under vacuum) in a very short time, about 5–15 s, in order to collect the required amount of gas for dating originating from rapidly dissolving (in comparison to lime aggregate) lime binder. The mortar samples prepared for GPC, due to the larger amount of sample required for a measurement and with respect to different methods of preparation, are represented both by the separated binder of different fractions (45–100, 80–100 μm) and by whole samples of plasters and mortars, which did not contain carbonate aggregate. To eliminate carbonate aggregate from so large a sample, it was sieved using the sieving column (with apertures from 0.045 mm to >2 mm) after size reduction by delicate crushing. Crushing was applied to the fragment carefully selected under a binocular microscope. The delicately grinded fragments were poured and agitated with distilled water, consequently passing into suspension. Sieving was carried out both for the suspension and the remaining part of the sample. Subsequently, the material was dried at >100 °C. The outermost part of this fragment, which could have been subjected to atmospheric influences, was removed. In the GPC technique, the whole carbonate fraction was dissolved with 4% hydrochloric acid in the vacuum apparatus, whereas for AMS it was treated with orthophosphoric acid (about 95%), giving CO_2. In GPC, CO_2 was subjected to dating, while in AMS, CO_2 was converted into graphite.

RESULTS AND DISCUSSION

The ^{14}C dating results of lime mortars from the Hippos (Sussita) settlement are presented in Table 1. The samples were ^{14}C dated by both GPC and AMS, with calibration undertaken using OxCal v 3.10 (Bronk Ramsey 1995, 2001). When the information concerning the age of a sample is limited to isolated ^{14}C dates, simple calibration is all that is required. However, if there is more information available, it seems most sensible to incorporate it into the probability distributions calculated. Sample 10 was dated in 2 laboratories (Gd-12823, 1310 ± 45 BP and Poz-7417, 1245 ± 35 BP). Because these dates come from the same sample, we combined them using the R_Combine option in OxCal. The combination was checked for internal consistency by a χ^2 test, which confirmed that the dates are in agreement with each other (t = 1.3[5% 3.8]). In the case of the studied site, we also know from reliable geological sources (Ben-Avraham et al. 1990, 2005; Amiran et al. 1994; Ellenblum et al. 1998)

Table 1 ^{14}C dating results.

Sample name	Sample mass (g C)	Lab code	Dating technique	Type of dated material	$\delta^{13}C$ (‰)	^{14}C age (BP)	Archaeological context
Hip14	0.00019	Poz-5088	AMS	Charcoal	–42.9	2025 ± 80	Floor pour of channel exposed at the chancel area
70H	1.5	Gd-12830	GPC	Carbonate binder	–11.45	1645 ± 35	Passage between the main and northern aisle; North-West Church (NWC)
Hip2/ch	0.00013	Poz-5087	AMS	Charcoal	–42.4	1570 ± 70	Collection pool in the agricultural installation to the south of the diakonikon
61Hip	0.00237	Poz-16078	AMS	Carbonate binder (separated basaltic aggregate covered with a film of binder)	0.2	1490 ± 30	Facade of the pastophorium northern wall
Hip2	0.00064	Poz-5016	AMS	Carbonate binder	–10.8	1295 ± 30	Collection pool in the agricultural installation to the south of the diakonikon
A1	0.3	Gd-17381	GPC	Carbonate binder, fraction 45–100 µm	–14.45	1140 ± 130	North-East Church (NEC), plaster from the apse
61H	0.3	Gd-18388	GPC	Carbonate binder 80–100 µm	–7.41	1080 ± 100	Façade of the pastophorium northern wall
Hip10	0.00167	Poz-7417	AMS	Carbonate binder	–9.5	1245 ± 35	Southern aisle, by the balustrade, northern face; NWC
10H	1.5	Gd-12823	GPC	Carbonate binder	–10.35	1310 ± 45	Southern aisle, by the balustrade, northern face; NWC

that in AD 749 an earthquake occurred and the Hippos settlement never rose from the ruins. This event defines a terminus ante quem (TAQ); thus, no dated sample can be older than AD 749. We combined ^{14}C dates and the above-mentioned information using the option TAQ in OxCal. The results of the calibration are presented in Figure 5 and Table 2.

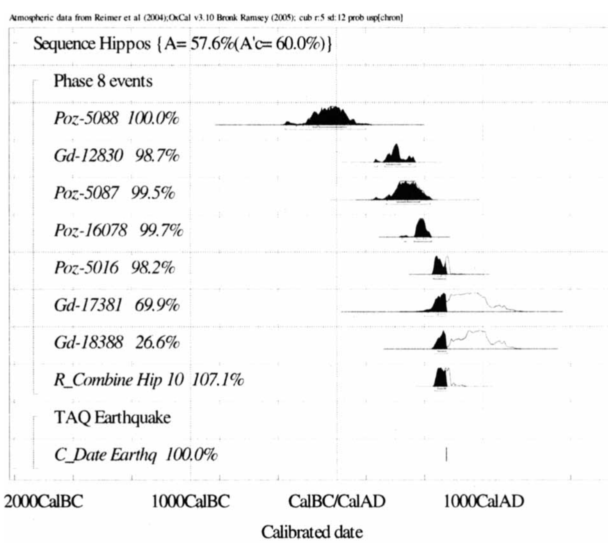

Figure 5 Results of calibration in graphical form

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Table 2 Results of calibration in numerical form.

Lab #	Date	Calibrated age (68.2% confidence interval)	Calibrated age (95.4% confidence interval)
Poz-5088	2025 ± 80	160 BC (5.4%) 130 BC 120 BC (62.8%) AD 70	350 BC (95.4%) AD 200
Gd-12830	1645 ± 35	AD 340 (64.2%) AD 440 AD 490 (4.0%) AD 510	AD 260 (2.1%) AD 280 AD 330 (93.3%) AD 540
Poz-5087	1570 ± 70	AD 410 (68.2%) AD 570	AD 340 (95.4%) AD 640
Poz-16078	1490 ± 30	AD 550 (68.2%) AD 610	AD 460 (1.4%) AD 480 AD 530 (94.0%) AD 650
Poz-5016	1295 ± 30	AD 665 (65.4%) AD 715 AD 740 (2.8%) AD 750	AD 660 (95.4%) AD 750
Gd-17381	1140 ± 130	AD 675 (68.2%) AD 750	AD 600 (95.4%) AD 760
Gd-18388	1080 ± 100	AD 690 (68.2%) AD 750	AD 665 (95.4%) AD 755
Poz-7417	1245 ± 35	AD 685 (58.4%) AD 730	AD 670 (95.4%) AD 750
Gd-12823	1310 ± 45	AD 735 (9.8%) AD 745	
R_Combine	1270 ± 28		

Dates from samples derived from the Sea of Galilee fit with the archaeological interpretation of the walls exposed during the excavations. The oldest dated sample (Hip14, Poz-5088, 2025 ± 80 BP) after calibration indicates a date in the range 2nd century BC to 1st century AD. As it comes from the reconnaissance in the main aisle of the NWC (functioned as the floor pour), archaeologists expect the date indicates the age of the Roman temple previously existing in place of the church. Sample Hip14 contained basaltic-carbonate aggregate that was finer than the rest of the samples. This mortar was characterized by strong porosity, the presence of 10–15% admixture of fine-grained (0.1–0.3 mm diameter) quartz sand, and contamination with charcoal particles. Therefore, the charcoal fragments were chosen for dating from this sample.

The results of dating indicate also 3 phases of the North-West Church (NWC) construction. When considered in the light of the 3 phases identified for the NWC by archaeologists, sample 70H (Gd-12830, 1645 ± 35 BP) might be assigned to the first phase of construction. The sample comes from the passage between the main aisle and the northern aisle of the NWC, from the pillar at the northern wall, and indicates the age of the church construction. It is a sample of pure lime plaster without aggregate. Sample 2 (Poz-5016, Poz-5087) is difficult to attribute to the second or third phase for the sake of probability distribution shape of this age after calibration (and what follows, a large width of confidence intervals). However, as it stems from archaeological premises, the first phase should rather be rejected. To the third phase of construction, samples Hip 2 (Poz-5016), 61H (Gd-18388), A1 (Gd-17381), and R_Combine for samples 10H (Gd-12823) and Hip10 (Poz-7417) can be assigned. The measurements conducted for the samples from the interiors of the NWC (sample 10) and the adjoining NEC (sample A1), both being inner finishing plasters, gave the same ages. The dating results of these 2 samples indicate the third chronological phase, the 7th–8th centuries AD. The obtained results suggest that the walls are contemporaneous. However, one should remember that while the age of mortars is closely bound with the construction age of a building, in the case of plasters the obtained age is not strictly connected with the time of building erection. Plasters may represent different phases of renovation or reconstruction and can be much younger in comparison to the age of the building. Therefore, their dating gives us information about the moment of applying the plaster on the wall, not about the time of the building erection—unlike what takes place for mortars, which are applied during the construction.

Some samples (e.g. sample 10) have been dated both by GPC (Gd-12823) and AMS (Poz-7417). Even despite different sample preparation (adjusted to the sample, but also respecting the method requirements, e.g. concerning sample size), they gave comparable results: Poz-7417, 1245 ± 35 BP; Gd-12823, 1310 ± 45 BP.

There is a puzzling discrepancy in the ^{14}C measurement results obtained by both techniques for sample 61. Sample 61 differs from the other samples dated because of the increased amount of carbonate aggregate. Thus, in order to verify the impact of this aggregate on the dating results, this sample was dated twice, each time prepared in a different way: 1) when the fragments of basaltic aggregate covered with a thin film of binder has been separated under the stereomicroscope, and 2) for the conventional technique, when the 80–100 μm fraction has been used in the measurement. Taking into consideration its composition and the manner of sample preparation, one should conclude that the AMS result, which is older than the age obtained by GPC, is overestimated. The film was thin that coated the aggregate grains after the previous delicate crushing of the mortar. However, the results (and their comparison with the GPC result for the fraction 80–100 μm) indicate that, most probably, the film was not completely clear of fine, "old" lime aggregate. In the case of the GPC dating, this aggregate had been separated by sifting out the coarser fraction. After sifting, only the fine fraction range of 80–100 μm was dated. Therefore, one infers that the real age of this sample is probably represented most closely by the GPC measurement (61H, Gd-18388, cal AD 690 [68.2%] AD 750, 68.2% confidence interval). Another important point is that the sample does not contain the traces of secondary recrystallization, which would cause the "rejuvenation" of the age; while for this sample the result obtained by the GPC technique is younger than the age given by the AMS technique for the aggregate fragment coated by thin layer of binder. However, to confirm this conclusion one may perform ^{14}C measurement for another sample from the same wall or date the same sample (61H) again. Another possibility would be to separate charcoal from that sample and compare the results. Further dating is ongoing as the excavation continues.

The charcoal from sample Hip14 came from its inner part, i.e. it was separated after grinding of the mortar. In the remaining samples, the carbonate binder has been dated, in bulk for the mortars devoid of carbonate aggregate (sample 10) and in the fraction <0.1 mm for the rest of the mortars (adequately to the observed composition). The result of ^{14}C dating obtained for the Hip14 charcoal sample is consistent with the wall stratigraphy, relative chronology (i.e. the sample was taken from the floor pour of channel exposed at the chancel area, from under a much younger floor of the later NWC), and with the archaeologists' estimations based on the historical data and excavation works. ^{14}C dating of the binder from this sample has not been carried out because of its much finer aggregate than in the other samples and because of the small size of this sample. In this case, charcoal was probably purposely added to the mortar in order to seal it and affect the desired properties of the floor pour. However, those fragments are already destroyed and after typical preliminary preparation they decompose—hence the small amount of carbon (Table 1, <1 mg) submitted for dating.

Sample Hip2 differed in that both the charcoal and the binder were dated. This sample contained singular, sporadic charcoal fragments (Hip2/ch, Poz-5087), which could represent accidental contamination during the mortar production. The dates obtained from the charcoal sample sometimes give ages that are too old. The main source of this error seems to be the old-wood effect. This hypothesis seems to be confirmed by the much younger age obtained from the measurement carried out for the binder from this sample (Hip2, Poz-5016). The samples were also carefully analyzed for possible secondary recrystallization. This sample was free of this influence.

For the binder samples dated by GPC and the charcoal dated by AMS, the bulk amount of the prepared sample was decomposed for measurement. For the carbonate material dated by AMS, the decomposition and gas collection was limited to the time interval 5–15 s, so as to eliminate possible dissolution of the harder, more resistant carbonate fragments of the aggregate.

When comparing the dating results presented in Table 1, the $\delta^{13}C$ values seem interesting, especially the very low values obtained for charcoal (–42‰). For the AMS dates, the $\delta^{13}C$ values were obtained after processing the samples for AMS measurements of ^{14}C content. These values were applied for the correction of the ^{14}C ages (Stuiver and Polach 1977). They do not indicate carbon fractionation in the process of mortar binding. Thus, low $\delta^{13}C$ values for the charcoal are connected with small size of the samples, below 1 mg of C (Table 1). Due to the small amount of analyzed material, it has not been identified to species. The wood fragments found in the NWC area were identified in the Wood Technology Institute in Poznań as most probably Lebanon cedar (Nawrocka et al. 2004).

For dates obtained using GPC (i.e. samples of greater mass), the $\delta^{13}C$ values were independently obtained from analyzing the part of the sample by mass spectrometry, and they indicate the amount of carbon isotopes fractionation in the process of mortar binding.

CONCLUSIONS

Carbonate binders have been dated in bulk for the samples devoid of carbonate aggregate (Hip10) and in the fraction <0.1 mm for the rest of the mortars. The results, obtained by GPC and AMS, are in agreement with the established archaeological stratigraphic sequence and enabled us to distinguish several phases of settlement development (especially the North-West Church [NWC] and the adjoining area).

Careful petrographic analysis was undertaken prior to dating. This included a detailed identification of particular components of the mortars, which is a very important prerequisite for reliable dating. Some parts of certain samples were dated by GPC initially, and then subsamples were dated using AMS to examine whether other methods of preparation had been sufficient to eliminate errors. This enabled us to decipher different stages of the expansion of Hippos from Hellenic to Byzantine times, indicated by the wall chronology.

The advantage of AMS is the very low sample size requirements. The selection of the dating method applied depends on the amount of material at our disposal. If we are not restricted by the size of the samples, the GPC technique can be also applied.

The results also show that ^{14}C dating of lime mortars, despite known difficulties (e.g. presence of carbonate aggregate), is possible and enables scientists to obtain ages for building construction after careful sample selection and a recognition of petrographic composition of binder and aggregate (possible exclusion of the samples that do not fit for dating and suitable preparation). The dates obtained from charcoal samples sometimes give ages that are too old (Hip2/ch, Poz-5087). The main source of this error seems to be related to the old-wood effect.

The choice of dating binders or charcoal fragments for dating should be made individually depending on the mortar composition, type, and size of the applied aggregate.

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