#### RESEARCH ARTICLE



# Development of an Interdisciplinary Approach to the Radiocarbon Dating of Earth Mortars from Alula Old Town (Saudi Arabia). Integration of Building Archaeology, Mortar Analysis and Radiocarbon Dating

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#### Abstract

This paper discusses the approach used to identify the most relevant chronological information on the historic development of the abandoned site of AIUla Old Town, in the northwest of the Kingdom of Saudi Arabia (Western Asia). The approach is based on the use of the radiocarbon method to date earth mortars samples and soil layers used to create the constructive sequence of some buildings and, in turn, the chronological evolution of the site. Eleven samples of organic material (i.e., charcoal and vegetable fibers) were carefully removed from mortar samples and soil levels from six buildings and structures in the northern and southern areas of the town. Buildings and soil layers were chosen for their stratigraphic relevance and conservation conditions, based on an initial archaeological analysis of both, buildings and underground structures. Laboratory-based mortar analysis led to the characterization of the mortar's inorganic fraction, and to the isolation of the organic material for the radiocarbon dating. Results from the accelerator mass spectrometry (AMS) laboratory provides evidence of a Late Mamluk/Early Ottoman constructive phase (i.e., 15th–17th c. AD) of the Old Town that was only partially known until very recently. Furthermore, the results allowed the identification of an Ottoman phase (i.e., 17th–19th c. AD) during which most of the buildings and structures were rebuilt, and of a Late Ottoman phase (i.e., 19th–20th c. AD) representing the most recent interventions before the end of the Ottoman occupation of the area.

#### Introduction

AlUla Old Town is an abandoned small town in the fertile oasis of Wādi al-Qurā, in the northwest of the Kingdom of Saudi Arabia, an area currently subject to an intense investigation and conservation program of its heritage (i.e., archaeological sites, still standing buildings, palm tree oasis). In this oasis, agricultural activities have been carried out for millennia thanks to the rich groundwater network that allows the intense cultivation of date palm (Battesti and Marty 2023) (Figure 1). The Wādi (also known as AlUla valley) is surrounded to the east and west by two mountain ranges. At the base of the west range was built AlUla Old Town, that stretches for about 7 hectares along the north-south axis of the valley (the modern town is a few kilometres south from it). Its original core was probably located around the small rock formation called *Umm Nāṣir* (Nasif 1988) that divides the settlement in a "north" and a "south" part, likely inhabited by two different leagues (the Shqîq league in the north and the Ḥilf league in the south, see Battesti and Marty 2023: 16–17). Before its abandonment in the 1980s, the Old Town



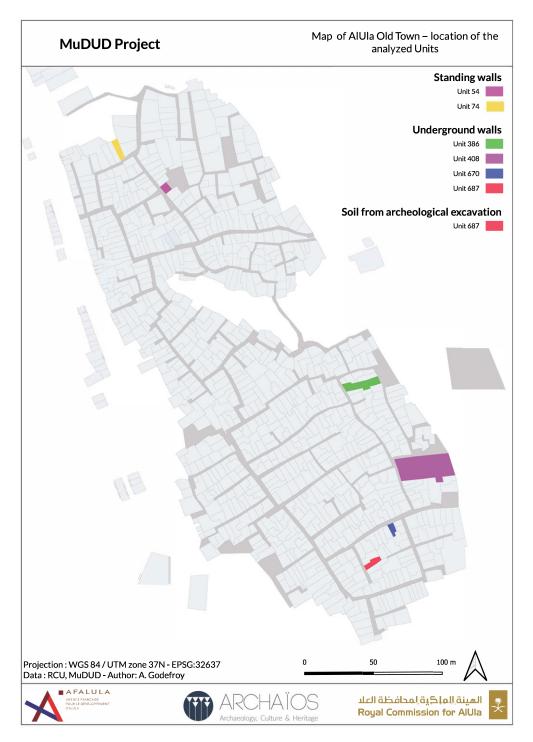
**Figure 1.** View of AlUla Old Town and of the oasis (centre) within the valley. The two mountain ranges protecting the wadi can be seen in the bottom-right corner of the image (west range) and in the top of it (east range). At the centre of the settlement, the Umm Nāṣir rock formation emerges from the buildings (picture by Piero Gilento, Archaïos).

had more than 900 buildings (Figure 2), mostly dwelling houses but also five mosques, a public bath, a school, and some open market spaces.

The first historic references to AlUla Old Town are in documents dating to the 8th c. AD (e.g., the name "AlUla" is in the list of places between Medina and Tabuk where the Prophet has offered prayers, as compiled by Ibn-Zubalah), where it is often associated to the other big settlement of the region: Al-Hijr (Arabic name of the UNESCO site of Hegra). In such documents, both sites are described as crossing points of the pilgrimage routes to Mecca. Other medieval sources from the 13th c. AD (e.g., a report of Abu Shamah indicates that the town had been settled by the early part of the 13th century under the rule of the Ayyubids) confirm the importance of AlUla as a passage for the convoys of pilgrims heading to Medina and Mecca. However, from the 16th to the 20th c. AD, the documents refer to AlUla not only in connection to its role in the journey to Mecca (e.g., descriptions from Al-Jaziri and Kibrit al-Madani), but also for the taxes due to the central government. Some documents refer to the construction activities carried out to improve the settlement's fortifications, with the aim of protecting it from the attacks of the nomads (a detailed list of sources, including those mentioned above, can be found in Nasif 1988, 135–137; Al-Muraikhi 2019). In the early 20th c. AD, the first dedicated railway station was constructed in AlManshiyah district just 3.5 km southeast of the settlement; this placed it on the important route connecting Damascus to Medina, with the consequent introduction of new construction materials and techniques such as lime mortars and sandstone ashlars. Overall, the written sources currently available for AlUla Old Town suggest the existence of an inhabited settlement from the 8th c. AD until the end of the 20th c. AD. However, such sources do not provide information on the size of the settlement, nor on its characteristics (Nasif 1988).

# The MuDUD Project

This lack of information led, in 2020, to the creation of the research project "Historical Investigation of AlUla Old Town – Multiscalar Documentation for Urban Dynamics" (MuDUD) that aims at investigating the evolution of the Old Town over the time. The project is carried out by the French archaeological company Archaïos and is funded and steered by the French Agency for the Development



**Figure 2.** Map of AlUla Old Town with the individual Building Units and, highlighted with colors, the Units discussed in this paper. The white space in the centre of the settlement is occupied by the Umm Nāṣir rock formation that divides the settlement into two parts (map by Anne Godefroy and Gabriela Camargo-Méndez, Archaïos).

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of AlUla (Afalula) in collaboration with the Royal Commission for AlUla (RCU). MuDUD is part of a wider research program that encompasses the entire valley and includes a number of scientific investigations in various sites, such as the "Dadan Archaeological Project" (Rohmer et al. 2020a, 2020b, 2022a, 2022b). All these activities aim at producing new chronological information (largely based on the use of the radiocarbon dating method) that, in the future, will allow the detailed and reliable reconstruction of the historic occupation of the valley. When completed, this reconstruction will be a unique reference for the entire Southwest of Asia.

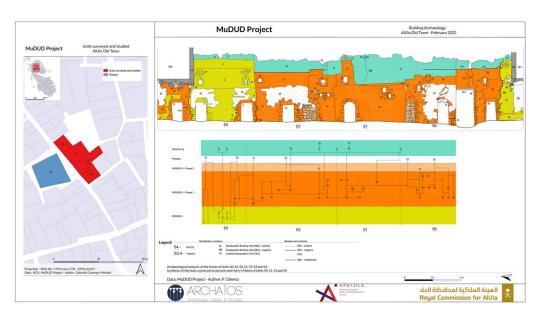
More specifically, MuDUD entails a variety of activities including archaeological excavations and the detailed analysis of the still standing buildings using the methods of Building Archaeology (Gilento 2020a, 2020b). Considering the limited archaeological record available for the Old Town, the use of radiocarbon dating as the main method to produce absolute chronologies was planned since the design stage and implemented throughout the project. To the best of our knowledge, to date, only a handful of individual radiocarbon dating have been attempted in AlUla Old Town, although no result is currently available in the public domain. Furthermore, none of the pre-existing chronological data was produced as a part of a large-scale investigation of the built heritage, or as a part of a structured investigation of the materials and technologies used for the construction of the settlement.

# Application of Building Archaeology Methods and Production of an Initial Chronological Sequence

Prior to the sampling for the radiocarbon dating, chosen groups of still standing buildings (i.e., Building Groups, or BG) in the north and south of the Old Town were subject to a detailed archaeological analysis using the methods of "Building Archaeology", based on the application of the stratigraphic principles to still standing buildings and structures. Underpinning this field of study is the detailed observation of the masonries (or parts of them) and the identification of their smallest homogeneous parts (i.e., Stratigraphic Building Unit, or SBU) and their respective relationships. To manage the complexity of the data emerging from such observations, a recording system that considers different "Reference Units" is adopted, as detailed in Brogiolo, Cagnana 2012. Like in traditional archaeological excavations, Building Archaeology makes use of the Harris' Matrix to provide a visual summary of the relative sequences of the SBU (i.e., before/after relationships), and to promote further analysis and interpretations of the data collected (Figure 3). The transformation of such relative sequences into absolute sequences is based on the use of specific chronological indicators such as *in situ* inscriptions, architectural decorations of known age, and—when possible—the results of archaeometric dating methods such as dendrochronology of timber elements, thermoluminescence of fired bricks, and the radiocarbon dating of mortars and plasters (Pesce et al. 2009, 2012).

In AlUla Old Town, the stratigraphic analysis of the buildings focussed on selected Units chosen for their accessibility, stratigraphic complexity, and the use of specific building techniques (Figure 2). This allowed the identification of three main construction periods that led to the production of an initial relative chronological sequence (subsequently modified based on <sup>14</sup>C results obtained) (Gilento 2020b, 143–145). The related periodization is reported below:

• Period 1 – to this period were attributed some wall sections of various façades mostly located in the northern area of the Town, like Unit 54 (Figures 3 and 4). Further Units presenting features related to Period I were also identified in the south and centre of the settlement. The stratigraphic analysis demonstrated that these façades were built before any adjacent building. Their constructive technique is characterised by the reuse of previously produced sandstone blocks, organised in very regular layers and laid on very thin mortar joints (1–1.5 cm). These characteristics are substantially different from those of the technique used in other buildings of the Old Town. Based on the stratigraphic relationships of these wall sections with the other buildings, it is possible to suggest a construction time preceding the beginning of the 20th c. AD.

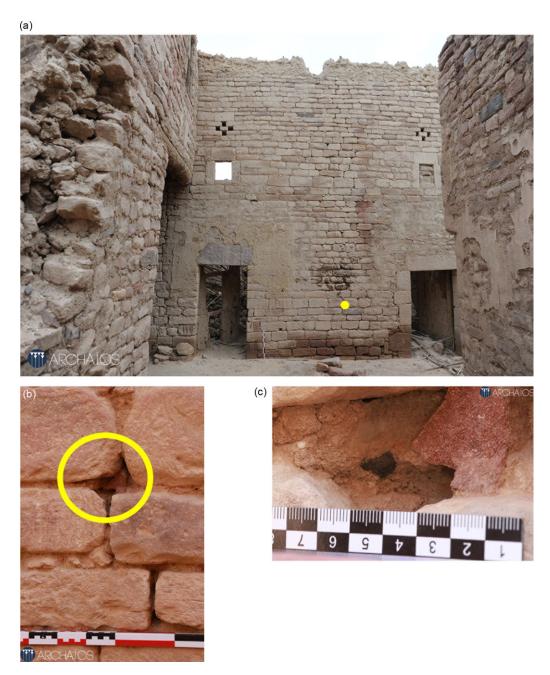


**Figure 3.** Stratigraphic reading and related Harris' matrix of U54, 53, 51 and 50 in the northern sector of the Old Town (analysis and drawing Piero Gilento, Archaïos; topographical location Gabriela Camargo-Méndez, Archaïos).

- Period 2 this period represents an important time for the development of the settlement since it denotes a substantial construction (or reconstruction) phase of the local built heritage. The construction activity that was identified in this period is likely to be related to a substantial urban expansion, clearly visible in the north-eastern part of the Town. The most representative units related to this period (e.g., U772, U787, U791 in the north part of the settlement, U631 and U635 in the south part) are characterised by a floor plan including an atrium around which other rooms are organised on one or two levels and, in some cases, by large terraces on the first floor. The building technique is based on the use of approximately dressed stone blocks specifically quarried for these buildings, laid on regular layers of 18/20 cm height. Door and window frames are made with finely worked stone blocks. Using the numerous inscriptions engraved in some of the door lintels, this period was dated between 1918 and the 1980s AD.
- Period 3 to this period were attributed all the traces of restoration activities carried out in the Old Town since the 1980s. No specific constructive technique can be associated to this period. However, unfired earth bricks or hand-shaped adobe elements were used in the reconstruction of the walls, whereas the metal rails originally used in the Ottoman railway were reused as horizontal elements (e.g., beams) in the buildings.

Beside the results emerged from the analysis of the still standing buildings and structures, some archaeological excavations, mostly concentrated in the southern part of the Town, brought to light several masonry structures (e.g., foundations), about 1 m below the ground level. The lack of physical connections (except in some very limited cases) with the still standing buildings and the different organization of the underground structures suggest the existence of an even earlier phase of use of the settlement that precedes the above-mentioned Period 1. Unfortunately, the pottery fragments found during the excavations could not be used for the absolute dating of these evidence because of the limited knowledge currently available on the historic pottery production in the region (although various studies are currently in progress to more accurately identify the chronologies of specific pottery productions). This led the archaeologists to extend the use of radiocarbon dating to the underground structures.

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**Figure 4.** General view from west of the façade of the U54 with the sampling point of the mortar (in yellow) (picture by Piero Gilento, Archaïos; b. Detail of the sampling point (picture by Giovanni Pesce, Northumbria University); c. Macrophotography of the charcoal sample embedded in the mortar (picture by Giovanni Pesce, Northumbria University).

#### Earth Mortars in AlUla Old Town

AlUla's built heritage was constructed using a variety of locally sourced materials: stone, earth, and timber. The timber from tamarind trees and date palm trees was mainly used for horizonal structures (e.g., floors and roofs), whereas stones and earth-based materials were mainly used for the vertical

structures (e.g., walls, foundations). Individual stone elements cut from the locally available yellow, red and violet sandstones were used in a variety of forms: from fully dressed ashlars to completely undressed pieces of stones. Earth-based building elements were used in the form of unfired earth bricks and balls of mud to be piled on the top of each other (i.e., the so-called Hand Shape Adobe; Houben 1994). To improve the properties of the locally poor soil, all earth-based materials were mixed with locally sourced organic and inorganic material such as straw (to improve cohesion and tensile strength) and crushed limestone concretion (likely to improve workability). Because of the limited durability of such materials, the heavy rains that occur in the valley once a year (albeit for a short period of time) have probably caused frequent loss of materials in the masonries (including their joints) that, therefore, had to be regularly repaired, as the local tradition suggests.

This paper presents the interdisciplinary methodological approach used to investigate the chronological evolution of the built heritage in AlUla Old Town. Within such context, building archaeology was used to identify the main development phases of the town; mortar analysis was carried out to provide a basic characterization of the mixes and define the materials and processes used in the mortar preparation at different stages, whereas the radiocarbon method was used to date the main phases. The selection of different materials or processes used in the construction of AlUla's built heritage at different times is essential, since it helps improving the current knowledge of the societies that built and lived in this settlement over the centuries. Indeed, changes in the local material culture could be related to a substantial change in the local society, to the influence exerted by an external constructive culture (e.g., arrival of builders from other areas), or to the exploitation of new locally available resources or the abandonment of exhausted ones.

Within the paper, six different historical times are mentioned. From the earliest to the latest these are: the Late Mamluk Sultanate (15th–16th c. AD), the Early Ottoman Empire (16th–17th c. AD), Ottoman Empire (18th–19th c. AD; time of the maximum expansion of this empire), the Late Ottoman Empire (19th–20th c. AD), the Modern Saudi Arabia (1920s–1980s AD), the Contemporary age (1980s AD–ongoing) (for specific historical and chronological references for the region, see Haykel 2010, 436–450; Lawson 2017; Masters 2013; Peskes 2010, 285–298).

# **Materials and Methods**

The mortar sampling was led by building archaeologists and carried out by mortar specialists, on the basis of the results of the archaeological analysis of the buildings. In AlUla Old Town, over 40 mortars samples were collected. From these, 6 samples from 6 Units (details in Table 1), in addition to one soil sample from Unit 687 were used for a detailed analysis.

The mortar characterization was carried out at Northumbria University's laboratories. Each sample was initially observed at low magnification (e.g.,  $10\times$ ) under a Leica MZ6 stereomicroscope. This allowed the identification and—when possible—the sampling of some of the most relevant elements of the mixtures (e.g., large aggregate particles, hair), including charcoal fragments and vegetable fibers. Subsequently, the samples (extremely crumbly) were gently hand-crushed and dry-sieved using meshes from 3.35 mm to 0.063 mm according to the BS ISO 11277:2020. This allowed for the assessment of the particle size distribution of the sandy/coarse fraction, and to separate the silty/clayey fraction for further analysis. The latter was used to determine the percentage of silt and clay by sedimentation using the pipette method according to the BS ISO 11277:2020, and to carry out X-ray diffraction analyses for the determination of the main clay minerals. X-ray analyses were carried out using a Rigaku StudioLab diffractometer. Quantitative analysis of various crystalline phases was obtained by applying the Rietveld refinement method using the tool provided by the Rigaku SmartLab Studio II software.

### Samples for the Radiocarbon Dating

Following the observation under the stereomicroscope of various mortar samples, 11 samples of organic material were isolated from the 7 mortar and soil samples subject to laboratory analyses, and

*Table 1.* List of organic samples sent to the laboratory for the <sup>14</sup>C determination with the related laboratory code and information on their origin and nature

Sample	Laboratory code	Unit number and type	Supposed chronology (AD)	Material	Mass (g)
MS_AU_10 (B) _CH01	Beta-642311	54	Ottoman empire	Charcoal	0.101
		(Standing wall)	(18–19 c.)		
MS_AU_10 (A) _VF01	Beta-657599		Ottoman empire (18–19 c.)	Vegetable fiber	0.022
MS_AU_27 (A) _CH02	Beta-642312	74 (Standing wall)	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.027
MS_AU_27 (A) _VF02	Beta-657600	-	Late Mamluk/Early Ottoman (15–17 c.)	Vegetable fiber	0.024
MS_AU_34 (A) _CH03	Beta-642313	386 (Underground wall)	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.006
MS_AU_34 (B) _CH04	Beta-642314	,	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.015
MS_AU_35 (A) _VF03	Beta-642315	687 (Underground wall)	Late Mamluk/Early Ottoman (15–17 c.)	Vegetable fiber	0.010
MS_AU_35 (B) _CH05	Beta-642316	,	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.027
CH_AU_36_CH06	Beta-657601	687 (Soil from archaeological excavation)	Late Mamluk/Early Ottoman (15–17 c.)	Vegetable fiber	0.148
MS_AU_40_CH07	Beta-642317	670 (Underground wall)	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.031
MS_AU_50_CH10	Beta-657604	408 (Underground wall)	Late Mamluk/Early Ottoman (15–17 c.)	Charcoal	0.024

individually sealed into plastic tubes to be sent to the Beta Analytics' AMS laboratory for radiocarbon dating. Table 1 reports the list of the samples sent to the AMS laboratory with the related information on the Unit where they were taken from, their supposed chronology (based on the archaeological analysis of the buildings and structures), the type of material (i.e., charcoal or vegetable fiber) and the related mass. As the table shows, of the 11 samples, 7 were made of charred material and 4 were non-charred vegetable fiber. When possible, 2 samples for each SBU were collected and dated to verify individual results. Particular attention was paid on-site and inside the laboratories to sample different types of organic material from the same SBU. This approach was used to assess the archaeological significance of the results obtained from the various materials. A detailed description of the SBU where the samples were collected and their relevance within the archaeological context is reported in the next paragraphs. As demonstrated by Table 1, all samples for the radiocarbon dating were within the recommended size (i.e., 5–100 mg). At the AMS laboratory, the samples were pretreated by washing alternatively in acid, alkaline and acid baths. This entailed a wash with HCl to remove the carbonates, followed by a wash with NaOH to remove the organic acid. The samples were, then, rinsed a final time with HCl to neutralize the NaOH. The samples were, subsequently, burned and turned into graphite, and then vaporized and run through the mass spectrometer.

#### Samples MS\_AU\_10 (B) \_CH01 and MS\_AU\_10(A)\_VF01 (Unit 54)

A charcoal sample (MS\_AU\_10(B)\_CH01) and a vegetable fiber (MS\_AU\_10(A)\_VF01) were collected from the façade of Unit 54 (Figure 4), in the northern area of the Old Town. This is the façade of a building overlooking a small open space in front of the northernmost mosque (U128). The façade was built using yellow-white sandstone elements reused from previous buildings, partially reworked and arranged in regular layers with rather thin mortar joints (1–2 cm thick). The stratigraphic analysis of the façade led to the identification of three construction phases within it (Figure 3). Both samples for the radiocarbon dating were taken from the mortar of the oldest part of the façade (BG9\_U54\_SBU0001). Results of the stratigraphic analysis suggested that this could be one of the oldest façades in the area, with a possible Late Ottoman (19th–20th c. AD) chronology.

# Samples MS\_AU\_27(A)\_CH02 and MS\_AU\_27(A)\_VF02 (Unit 74)

A charcoal fragment (MS\_AU\_27(A)\_CH02) and a vegetable fiber (MS\_AU\_27(A)\_VF02) were taken from the north-facing external wall of U74 (Figure 2). This Unit is located to the northernmost area of the Old Town, built on the top of an embarkment that was based on a large calcareous concretion probably used as a quarry for construction material at some point in time. The topographical location, structural grandeur (7 m high), and stratigraphic composition of the wall from which the sample was taken suggested the possibility that this Unit was part of a town wall. The stratigraphic analysis of the Unit highlighted several construction phases within it. Attention was paid during the sampling work to make sure that the charcoal and vegetable fibers were taken from its oldest part (BG3\_FU001\_F3\_SBU20409). The analysis of the wall suggested a possible construction time in the Late Mamluk - Early Ottoman period (i.e., 15th–17th c. AD).

# Samples MS\_AU\_34(A)\_CH03 and MS\_AU\_34(B)\_CH04 (Unit 386)

These two charcoal samples were taken from some masonry structures emerged from an archaeological excavation carried out by the RCU inside the Ibn Yunus Mosque (U386, north of one of the main roads in the southern part of the Old Town; Figure 2). At the time of the excavation, some charcoal samples were collected from various soil levels to radiocarbon date the structures. However, little is known about this archaeological excavation and the interpretation of the <sup>14</sup>C results. Hence, a new study of the same

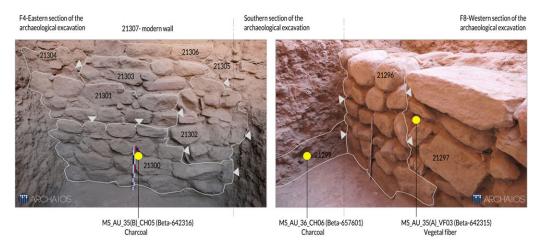


**Figure 5.** Stratigraphic analyses of the masonry walls found in the Ibn Yusun mosque with the related sampling points for the charcoal fragments collected: (left) U386 and (right) U670 (Pictures and elaboration by Piero Gilento, Archaïos).

archaeological sections and structures was carried out within the MuDUD project to elucidate their stratigraphic sequences and clarify their existence within the rest of the mosque. As a part of this activity, two new charcoal samples were collected from the two stratigraphically oldest structures found in the excavation. The charcoal sample MS\_AU\_34 (A) \_CH03 was embedded in the mortar of a wall with north-south orientation (U386\_FUA\_F8\_SBU21280) at the edge of the eastern section of the excavation. The charcoal sample MS\_AU\_34 (B) \_CH04, instead, was taken from the mortar of the wall U386\_FUA\_F9\_SBU21282, perpendicular to the previous one. The stratigraphic analysis determined that the SBU21280 was built before the SBU21282 (Figure 5, left). Building Archaeology specialists suggested a sequence where the oldest structures (U386\_FUA\_F8\_SBU21280 and U386\_FUA\_F9\_SBU21282) could be of Late Mamluk–Early Ottoman origin (i.e., 15th–17th c. AD).

# Sample MS\_AU\_40\_CH07 (Unit 670)

Unit 670 is in the southernmost part of the Old Town and is part of BG34 (Figure 2). The sample collected in this Unit was taken from an archaeological excavation carried out in the rear room of the Unit (U670\_FUB), where an east-west orientated wall (U670\_FUB\_F6) was found in the north section. SBU21291 was made of undressed stone elements arranged in irregular layers with large mortar joints filled with wedges (likely the foundation of a wall built using earth-based elements). From this SBU a charcoal fragment (MS\_AU\_40\_CH07) was collected (Figure 5, right). The excavation found soil



**Figure 6.** Images of the masonry walls emerged from the test trench excavated inside the Unit 687 with the sampling points of the organic material collected: (left) east section of the trench with the location of the charcoal sample MS\_AU35(B)\_CH05; (right) west section of the trench with the sampling points of the charcoal sample MS\_AU\_36\_CH06 and of the vegetable fibre MS\_AU\_35(A)\_VF03 (Pictures and elaboration by Piero Gilento, Archaïos).

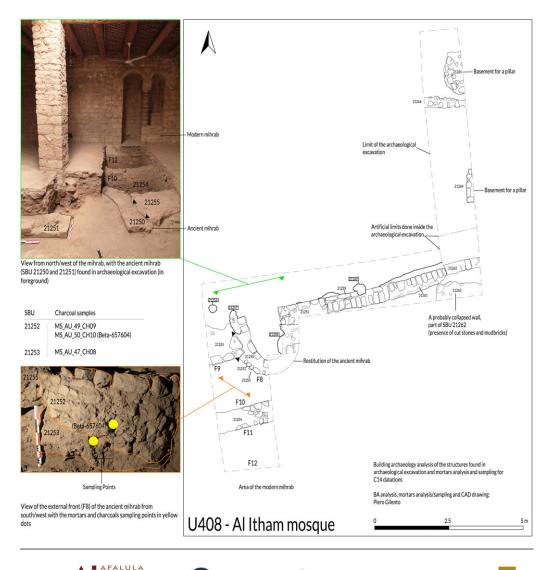
layers rich in pottery that has preliminarily been dated between the 6th and the 10th c. AD (although these are only initial chronological hypothesis). Hence, the radiocarbon dating of this wall was considered essential to better define the chronology of the entire settlement. Considering the condition of the structure and its position, a preliminary chronology placed the construction of this wall in the Late Mamluk – Early Ottoman period (i.e., 15th–17th c. AD).

# Samples MS\_AU\_35 (A) \_VF03, MS\_AU\_35 (B) \_CH05 and CH\_AU\_36\_CH06 (Unit 687)

U687 is a rather long rectangular dwelling house in the centre of BG37, not far from U670 mentioned above (Figure 2). A test trench was opened within a small space in the easternmost part of the Unit (U687\_FUC). From this excavation, a rich archaeological stratigraphy emerged that included two masonry structures (U687\_FUC\_F4 and U687\_FUC\_F8) running parallel to each other, with no physical connection. The structures were analysed, and a mortar sample was taken from each of them: from SBU21297 in U687\_FUC\_F8, and from SBU21300 in U687\_FUC\_F4. The laboratory analysis of these mortars allowed the collection of, respectively, a vegetable fiber (MS\_AU\_35(A)\_VF03) and a charcoal sample (MS\_AU\_35(B)\_CH05; respectively Figures 6 right and 6 left). Another charcoal sample (CH\_AU\_36\_CH06) was taken from a layer of soil (SU21299) extremely rich in charcoal fragments, located in the lowest part of the southern end of the excavation (Figure 6, right). The archaeological analysis of the two walls suggests a Late Mamluk–Early Ottoman origin (i.e., 15th–17th c. AD), or an even older origin.

# MS\_AU\_50\_CH10 (U408)

U408 corresponds to Al-Itham Mosque, which is the last congregational mosque in AlUla Old Town (Figure 2). This building is located on the east edge of the southern area of the settlement and is made of just a single praying room 37 m long and 13 m wide, with an east-west orientation. When the mosque was subject to some conservation works in 2022, archaeological excavations were carried out by an archaeological company. The largest of these excavations was in the centre of the room, in front of the





**Figure 7.** Plan of the excavation inside the Al-Itham mosque (right-hand side) and images of the sounding (left-hand side). The image on the top left shows the relationship between the current and old mihrab of the mosque, whereas the image on the bottom left shows the SU where the dated sample was collected (Analysis, pictures and CAD drawing by Piero Gilento, Archaïos).

mihrab (the apsidal space indicating the direction to Mecca; Figure 7, top left). Inside this trench several masonry structures emerged, that were subsequently subject to documentation and historical analysis by the MuDUD project. Because of its shape, orientation and position, one of these structures was interpreted as the remains of an older mihrab. The external side of this wall was built using undressed stones laid on irregular layers and bonded by thick mortar joints that allowed the collection of the charcoal sample MS\_AU\_50\_CH10 from U408\_FUA\_F8\_SBU21252 (Figure 7, bottom left). Archaeological evidence suggested a construction time between the 15th and the 16th c. AD.

#### Results and Discussion

# Mortar Analysis

Observations of the mortar samples under the stereomicroscope led to the identification of 4 ingredients that strongly characterise the mortars: concretion fragments of various diameter (likely from a quarry at the northern edge of the settlement), vegetable fibers, animal hair, and charcoal fragments. As shown in Table 2, three of the 7 mortar samples that were used for the radiocarbon dating did not contain vegetable fibers, whereas all of them contained charcoal fragments (the sample from U670 was not characterised because collected during an archaeological activity that took place after the laboratory analyses were concluded). Furthermore, 5 samples did not contain hair but contained concretion fragments (only the sample from U54 did not contain these fragments but contained animal hair).

The dry sieving of the samples led to the identification of 3 groups of mortars with different percentage of particles with a diameter above 3.35 mm (i.e., the largest mesh used): group I is the group of samples with 70–90% particles above the sieve limit; group II.A includes samples with 35–45% of particles with diameter >3.35 mm, and Group II.B includes samples with 5–25% of particles with diameter >3.35 mm. As shown in Table 1, all mortars containing organic material were part of the Group II.B (with the smallest percentage of large particles).

Results of the analysis on the silt and clay fractions proved that all mixtures were very poor in silt and clay: the sample containing the highest percentage of clay produced a result of only 3%. Results of the XRD analyses for the silt/clay fraction showed that the finest fractions in all mortars consisted of: quartz (with percentage varying between 4% and 70%), calcite (6%–61%), clay minerals (1%–40%) mainly represented by kaolinite and illite (however, sample MS\_AU\_30 also contained traces of sepiolite), and hematite (1%–14%). Dolomite was detected only in sample MS\_AU\_36 (i.e., mortar sample from which charcoal sample CH\_AU\_36\_CH06 was extracted), whereas halite only in sample MS\_AU\_34(A). Gypsum was detected only in traces (2–6%) in samples MS\_AU\_36 and in sample MS\_AU\_34(A/B) (11-35%).

Overall, the mortar analysis suggested a rather similar composition for all samples bearing organic material. All mortars have a very low clay content (i.e., a modern earth mortar requires a clay content

**Table 2.** List of the mortar samples analysed with the related topographic location, and some of the results from the laboratory characterization (i.e., particle size distribution of the aggregate—PSD—grouping based on the percentage of silt/clay content, record of the presence of vegetal fibers, animal hair, charred material, and concretion lumps)

Unit	Mortar sample	Area	Group by PSD	Vegetable fibers	Animal hair	Concretion lumps	Charred material
54	MS_AU_10(B) CH01	North	II.B	Yes	Yes	No	Yes
74	MS_AU_27(A) _CH02	North	II.B	Yes	No	Yes	Yes
386	MS_AU_34(A) _CH03	South	II.B	No	No	Yes	Yes
386	MS_AU_34(B) CH04	South	II.B	No	No	Yes	Yes
687	MS_AU_35(B) CH05	South	II.B	No	No	Yes	Yes
687	MS_AU_35(A) VF03	South	II.B	Yes	No	Yes	Yes
670	MS_AU_40_CH07	South	_	_	_	_	_

between 10% and 40%; Henry et al. 2015), and—considering some of the ingredients used (i.e., the concretion fragments)—it is likely that these were produced using only locally sourced materials.

#### Radiocarbon Dating

The results obtained from the AMS laboratory are presented in Table 3 and in Figure 8, where they are quoted in accordance with the Trondheim Convention (Stuiver and Pearson 1986) as conventional <sup>14</sup>C ages (Stuiver and Polach 1977). The calibrated date ranges were calculated using the IntCal20 calibration curve of Reimer et al. (2020) and BetaCal 4.20. Calibrations are cited in the table and in the text with the related confidence intervals.

Overall, the results can be divided in 3 main chronological groups:

- The first group includes the samples MS\_AU\_10(B)\_CH01, MS\_AU\_34(B)\_CH04 and MS\_AU\_50\_CH07 dated between the 15th and the 17th c. AD. However, whereas the first sample was taken from a still standing wall, the other two samples were removed from walls found in archaeological excavations.
- The second group includes two vegetable samples removed from standing walls (MS\_AU\_10(A) \_VF01 and MS\_AU\_27(A)\_VF02) and a charcoal sample taken from a layer of soil during an archaeological excavation (CH\_AU\_36\_CH06). Results of the radiocarbon dating suggest an age between the end of the 17th and the beginning of the 20th c. AD.
- The third group includes 3 charcoal samples MS\_AU\_27 (A)\_CH02, MS\_AU\_34 (A)\_CH03, MS\_AU\_35 (B)\_CH05, MS\_AU\_50\_CH10, and a vegetal fiber (MS\_AU\_35 (A)\_VF03) all dated to a period between the 16th and the 20th c. AD (however a higher probability relates to a period between the 16th and the 17th c. AD).

A more detailed analysis of the results obtained based on the SBU where the samples were taken is reported in the following paragraphs.

# Units 54 and 74—Radiocarbon Dating of Charcoal and Vegetable Fibers

Different results were obtained for the charcoal and the vegetable fiber collected in the mortar of Unit 74 (colored in yellow in Figure 8). The charcoal sample MS\_AU\_27 (A)\_CH02 was dated to 1618–1670 AD (45.3%) and to 1508–1594 AD (42.7%), whereas the vegetable fiber sample MS\_AU\_27 (A) \_VF02 was dated to 1810–1918 AD (67.8%) and to 1694–1726 AD (27.6%). Similarly to this unit, different results were also obtained for the charcoal sample MS\_AU\_10(B)\_CH01 and the vegetable fiber MS\_AU\_10 (A) \_VF01 taken from the façade of Unit 54 (colored in pink in Figure 8). The charcoal sample was dated to 1474–1638 AD (95.4%), while the vegetable fiber was dated to 1800–1908 AD (68.6%) and 1682–1738 AD (25.7%).

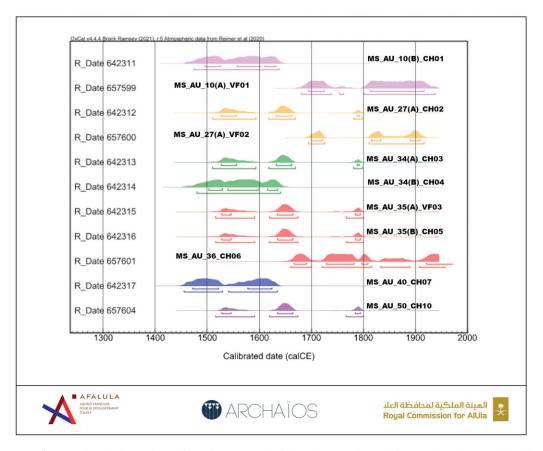
Various hypotheses were considered to explain such difference between the results of the charcoal samples and those of the vegetable fiber. A hypothesis that was initially considered was the possibility that the charcoal samples represented the actual construction time of the buildings, whereas the vegetable fiber a later maintenance work. However, a careful analysis of the stratigraphy, of the photographs taken during the sampling work, and of all radiocarbon data, did not support such difference in the origin for the dated materials. Another hypothesis considered the <sup>14</sup>C results of the vegetable fibers as representing the actual construction time of the buildings, whereas the charcoal samples as a contamination due to the accidental embedding of older material in the mortars at the time when these were prepared. A further hypothesis entailed the "old wood" problem (Schiffer 1986), although in desertic area like in the AlUla region is less relevant due to the limited lifespan of species like tamarind and palm. After careful evaluation of all data, the first hypothesis was disregarded,

Table 3. Results of radiocarbon dating

Sample	Laboratory code	Context	Supposed chronology	Material	$\delta^{13}$ C‰ PDB	Conventional <sup>14</sup> C age BP	Calibrated date (2σ) cal AD
MS_AU_10 (B) _CH01	Beta-642311	Unit 54 Standing wall	Ottoman Empire (18–19 c.)	Charcoal	-24.8	340 ± 30	(95.4%) 1474–1638
MS_AU_10 (A) _VF01	Beta-657599	Unit 54 Standing wall	Ottoman Empire (18–19 c.)	Vegetable fiber	-12.4	110 ± 30	(68.6%) 1800–1908 (25.7%) 1682–1738 (1.2%) 1754–1762
MS_AU_27 (A) _CH02	Beta-642312	Unit 74 Standing wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-22.9	270 ± 30	(45.3%) 1618–1670 (42.7%) 1508–1594 (7%) 1780–1798 (0.4%) 1946–post 1950
MS_AU_27 (A) _VF02	Beta-657600	Unit 74 Standing wall	Late Mamluk/ Early Ottoman (15–17 c.)	Vegetable fiber	-23.2	50 ± 30	(67.8%) 1810–1918 (27.6%) 1694–1726
MS_AU_34 (A) _CH03	Beta-642313	Unit 386 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-26.2	270 ± 30	(45.3%) 1618 - 1670 (42.7%) 1508- 1594 (7%) 1780-1798 (0.4%) 1946-post-1950
MS_AU_34 (B) _CH04	Beta-642314	Unit 386 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-26.1	330 ± 30	(95.4%) 1480–1640
MS_AU_35 (A) _VF03	Beta-642315	Unit 687 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Vegetable fiber	-24.4	260 ± 30	(51.5%) 1620– 1674 (28.7%) 1516– 1590 (13.6%) 1766– 1800 (1.6%) 1942–post 1950
MS_AU_35 (B) _CH05	Beta-642316	Unit 687 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-20.1	260 ± 30	(51.5%) 1620– 1674 (28.7%) 1516– 1590 (13.6%) 1766– 1800 (1.6%) 1942– post 1950
CH_AU_36_CH06	Beta-657601	Unit 687 Soil from	Ottoman Empire (18–19 c.)	Charcoal	-22.7	170 ± 30	(46.%) 1720–1815 (19.5%) 1907– post-1950

 Table 3. (Continued)

Sample	Laboratory code	Context	Supposed chronology	Material	δ <sup>13</sup> C‰ PDB	Conventional <sup>14</sup> C age BP	Calibrated date (2σ) cal AD
		archaeological excavation					(17.4%) 1660–1700 (12.5%) 1832–1890
MS_AU_40_CH07 Be	eta-642317	Unit 670 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-11.4	360 ± 30	(49.9%)–1540– 1634 (45.5%) 1456– 1529
MS_AU_50_CH10 Be	eta-657604	Unit 408 Underground wall	Late Mamluk/ Early Ottoman (15–17 c.)	Charcoal	-22.1	260 ± 30	(51.5%) 1620–1674 (28.7) 1516–1590 (13.6) 1766–1800 (1.6%) 1941–Post 1950



**Figure 8.** Results of the radiocarbon dating method for the samples of charcoal and vegetable fibre analysed in this research. The different colours represent different SBUs where the samples were taken: pink=U54; yellow=U74; green=U386; red=U687; purple=U408 (same colours are used in Figure 2) (Elaboration by Maureen Le Doaré and Piero Gilento Archaïos).

because the results obtained are in good agreement with the results of the stratigraphic analysis of the building that suggested a possible construction time between the 18th and the 19th c. AD for Unit 54.

Consequence of this interpretation is that the buildings where these mortar samples were collected can be dated to the Ottoman time (i.e., 18th–19th c. AD). In this case, it is likely that the façade of U54 remained intact over the centuries because of the good quality of the building technique and, possibly, of the "prestige" of the building itself (as also suggested by its location in a very central part of the northern area of the Old Town). If this is the case, the façade can, then, be used as a "fossil guide" for the study of the masonry techniques and typologies (doors and windows) used in AlUla Old Town during the Ottoman period (Bonnal et al, forthcoming).

It is important to stress that, differently from the case of Unit 54 (where the initial chronological hypothesis was confirmed by the results of the radiocarbon dating), the initial suggested construction time for the Unit 74 is between the 15th–16th c. AD was not supported by the results of the radiocarbon dating and, therefore, the initial suggestion was reconsidered by the archaeologists.

### Ibn Yunus Mosque: Stratigraphic Sequence and Radiocarbon Results

The two charcoal samples MS\_AU\_34(A)\_CH03 and MS\_AU\_34(B)\_CH04 (colored green in Figure 8) were taken from two walls found in the archaeological excavation inside the Ibn Yunus

Mosque (U386). Their stratigraphy clearly suggested an older age for the wall with north-south orientation compared to the one with east-west orientation. This sequence is confirmed by the radiocarbon dating results that dates the north-south wall to  $330 \pm 30$  BP whereas the east-west wall to the  $270 \pm 30$  BP. The fact that the two results belong to the same historic period confirms that in the Late Mamluk–Early Ottoman period (i.e., 15th–17th c. AD), some structures already existed in the area currently occupied by the mosque. Very little is known about these structures and their functions since they are only marginally preserved above the foundation level. A clear indication that emerges from these structures is that, at that time, the buildings had a different orientation compared to the mosque currently visible.

# Al-Itham Mosque: An Early Ottoman Building in the Southern Area

Some masonry structures including a mihrab emerged inside one of the soundings within the Al-Itham mosque (U408). The mihrab found in the excavation is almost aligned with the one currently in use. The charcoal sample MS\_AU\_50\_CH10 (colored purple in Figure 8) taken from its external side was dated to 1620–1674 AD (51.5%) and to 1516–1590 AD (28.7%). Both results (making a total probability of 80%) are within the Early Ottoman period (16th–17th c. AD). A later chronological period making an overall probability of about 15% suggests a later period: 1766–1800 AD (13.6%) and 1941-Post 1950 (1.6%). However, considering the conditions of the structures where the sample was collected and its context, it is most likely that the mosque was built in the Early Ottoman period (16th–17th c. AD) and this allow inferring that at this time a mosque did exist in the southern area of the Old Town.

#### **Unit 687**

Both masonry structures found in the archaeological excavation of U687 have been dated to the same Ottoman period (17th c. AD) using the charcoal sample MS\_AU\_35(B)\_CH05 and a vegetable fiber MS\_AU\_35(A)\_VF03 (colored red in Figure 8). This is the only case where different materials taken from separate structures were dated to the same period. All data related to these masonries (*i.e.*, the archaeological context, the building technology used, and the mortar analysis) confirm a homogenous "constructive culture" for the two structures, which supports the results of the radiocarbon dating.

Furthermore, the result of the additional charcoal sample (CH\_AU\_36\_CH06) collected within U687 supports the chronology obtained for the two masonry structures. Indeed, this charcoal sample was taken from a layer of soil that had a clear stratigraphic relationship with the masonries, having formed later. The charcoal sample from this layer was dated to 1720–1815 AD (46%), 1907-Post 1950 AD (19.5%), 1660–1700 AD (17.4%), 1832–1890 AD (12.5%). Overall, this is the only sample dated to the Ottoman Period (18th–20th c. AD) among all samples dated in the south area of the Old Town, and its chronology is in good agreement with the chronology of the two vegetable fibers dated in the north area of the Old Town discussed earlier.

#### **Unit 670**

Despite having been found in association with a large amount of pottery fragments, little is known about the masonry structure unearthed in the archaeological excavation opened inside Unit 670. However, the charcoal sample MS\_AU\_40\_CH07 (colored blue in Figure 8) taken from a section of the structure was dated to 1540–1634 AD (49.9%) and 1456–1529 AD (45.5%). The first chronological range is within the Early Ottoman Empire (16th–17th c. AD), whereas the second one spans across the Late Mamluk Sultanate and the Early Ottoman Empire (15th–17th c. AD). Considering the probabilities emerged from the analysis, it is difficult to identify the most likely period of construction but it is interesting to note that the second chronological range is in good agreement with one of the two chronological groups

already identified (i.e., the Late Mamluk-Early Ottoman period). If confirmed, this sample might play an important role in the study of the pottery fragments.

#### Discussion

Considering the results obtained and the topographic position of the buildings and structures analysed, it is possible to suggest that two important times related to the history of the Town are represented in these results: the first period dates between the mid-15th c. AD and the second half of the 17th c. AD and corresponds to a Late Mamluk-Early Ottoman period, whereas the second period is between the end of the 17th c. AD and the 1920s AD, and corresponds to the Ottoman period.

The first of the two periods is mainly represented in the masonries found inside archaeological excavations in the southern area of the settlement, below the current ground level. This means that, between the end of the Mamluk Sultanate and the beginning of the Ottoman Empire, the southern area of the Old Town was already built, although different buildings existed compared with those visible nowadays. This part of the settlement included a mosque and other structures (likely dwelling houses) that had a different orientation compared to the current settlement, since the road system was based on wider alleys with different orientation compared to the current one. Probably in the Ottoman period, this area of the Town was substantially rebuilt acquiring the characteristics we see nowadays.

The few radiocarbon data related to the northern area provide further information on the settlement in the Ottoman period, since they confirm that between mid-15th and mid-17th c. AD the Old Town covered an area approximately similar to the one currently occupied, except for the north-eastern part of the settlement that was probably built between 1918 and the 1980s (i.e., phase 3 mentioned above). It is important to stress that constructive activities carried out in the 17th c. are known from a written source (Ḥājjī Khalīfah) documenting the restoration of the stronghold and its fortifications for protection against the Bedouins raids (Nasif 1988).

The second major constructive period highlighted by the radiocarbon results, between the end of the 17th c. and the 1920s, is a rather long historic period during which most of the still standing buildings were built. Some of these buildings are of high technical quality (at least for the façades) and are still now in rather good conditions.

Considering the very consistent results of all the mortar analysis, the consistency of the radiocarbon dating and the widespread distribution of the buildings analysed, it is worth considering the possibility that the soil used to produce all mortars was sourced in a very specific area of or around the town, and that such area was used regularly for the supply of construction materials over the centuries.

From a methodological perspective, this research proves that the usability of the chronological information obtained from the radiocarbon dating of organic material embedded in earth mortars is strictly related to two factors: (1) a correct sampling work (e.g., that pays particular attention to the presence of repairing mortars), and (2) a correct and detailed stratigraphic analysis of the buildings and structures where the samples are taken (e.g., to identify the correct sampling points). In such context, the MuDUD project represents a reference point in the investigation of the built heritage of the Arabian Peninsula.

#### Conclusions

This paper discussed the results of the radiocarbon dating of 11 samples of organic material (i.e., charcoal and vegetable fibers) taken from 7 mortar samples from soil level and structures in AlUla Old Town, with the aim of improving our knowledge of the history of the settlement and acquiring a better understanding of the still standing buildings. The main challenge faced by the archaeologists in AlUla Old Town was the creation of a verifiable, coherent, and reproducible dating tools like the typology of the masonry techniques that can be used on a large scale such as entire settlements. To create such tool, this project focussed on the radiocarbon dating of the organic materials embedded in

the mortars used for the construction of buildings and structures (for which a detailed archaeological analysis of the buildings, a laboratory characterization of the mortars, and their radiocarbon dating were necessary). The presence of material of various ages embedded in the mortars led to the identification of the vegetable fibers as the most accurate chronological indicators of the construction time of the buildings. However, the results of this research demonstrate that, if the radiocarbon dating of charred material is used within precise stratigraphic contexts and coherent datasets, it can still bear some useful information, as proved by the results of Unit 687.

Overall, from the results obtained emerges that most of the buildings currently visible above the ground in AlUla Old Town were constructed during the late Ottoman period (19th–20th c. AD), and that an earlier settlement dated between the end of the Mamluk Sultanate and the beginning of the Ottoman Empire (15th–17th c. AD) likely existed in the same location although is currently preserved only in some structures underground.

To conclude, it is important to stress that these results are only preliminary, and the analysis of further samples are needed to reconstruct the chronological development of the entire settlement. As this research confirms, independently from the number of samples analysed, the results of the radiocarbon dating of any sample should be used in conjunction with other tools such as the archaeological analysis of the buildings that can be used to validate the results obtained (e.g., Unit 54), or as a method to question and deepen our understanding of the historic events that affected a building or a structure (e.g., reparation works) when in disagreement (e.g., Unit 74).

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