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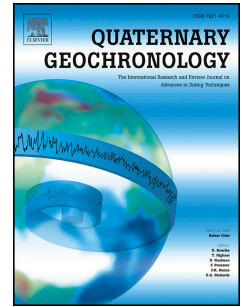
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Forgotten news: Shellfish isotopic insight into changing sea-level and associated impact on the first settlers of the Mariana Archipelago

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Forgotten News: Shellfish isotopic insight into changing sea-level and associated impact on the first settlers of the Mariana Archipelago.

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## Abstract

The colonisation of the Pacific is an important chapter in human dispersal for which chronological control is primarily provided by radiocarbon ( $^{14}\text{C}$ ) dates. In this context, the ability to reliably date shellfish is important because alternative dating materials, such as charcoal and bone, are typically highly degraded. However, the interpretation of shell  $^{14}\text{C}$  results is not always black and white because  $^{14}\text{C}$  is not evenly distributed throughout the marine environment, with estuarine taxa more likely to incorporate terrestrial sources of carbon. Regions where water has percolated through limestone bedrock provide an additional problem since ancient carbon is introduced into the estuarine waters. This “hardwater” has been put forward to explain old 3500 cal. BP results from culturally significant shells recovered from the site of Unai Bapot (Bapot-1) on the island of Saipan (Petchey et al. 2017). While arguments for (Carson and Hung 2017) and against (Rieth and Athens 2017) early settlement dates remain polarised, little attention has been given to the idea of change in the marine  $^{14}\text{C}$  reservoir over time, or to possible species-specific offsets in shell  $^{14}\text{C}$ .

In this paper, we further develop a tri-isotope approach using  $^{14}\text{C}$ ,  $\delta^{13}\text{C}$ ,  $\delta^{18}\text{O}$  to identify carbon source. To investigate which shellfish are more prone to erroneous ages we have selected shell taxa that cover a range of nearshore environments commonly found in Pacific archaeological sites; including *Anadara antiquata*, *Gafrarium pectinatum* (both estuarine) and *Tridacna* (marine/reef). To test the possibility of change over time we extend the dating of the site beyond the earliest occupation layers to deposits considered to post-date the end of the mid-Holocene drawdown in sea-level.

Keywords: Reservoir offset; Radiocarbon dating; Hardwaters; Mariana Islands; Colonisation

## 1. Introduction

The exact age of first colonisation of the Mariana Islands (Figure 1) is debated, with arguments split between early (pre-3500 years BP; Hung et al. 2011, Carson 2014, Carson and Hung 2017) and late (3200 years BP; Petchey et al. 2017, Rieth and Athens 2017) hypotheses. This duality hinges entirely on disparate interpretations of the same radiocarbon dataset, in particular, whether early shell dates from the site of Unai Bapot (Bapot-1) are, or are not, affected by hardwater. The younger age has significant implications for cultural development throughout the region (Rieth and Athens 2017, Fitzpatrick and Jew 2018) because the oldest movement into Western Micronesia and Lapita<sup>1</sup> movement into the West Pacific are now thought to occur at a similar time; this, in turn, suggests a very rapid dispersal of people through these regions – within 200 years. It also raises the possibility that the formative years of Lapita in the Bismarck region are even later than currently thought, which has consequences for theories about cultural development in that region and beyond (Montenegro et al. 2016, Rieth and Athens 2017). Moreover, recognised as possibly the longest ocean voyage of its time (over 2000 km) we are no closer to understanding why these seafaring people subsequently and inexplicably remained in isolation in the Mariana's for more than 2000 years (Hung et al. 2011; Vilar et al. 2012; Fitzpatrick and Callaghan 2013). During this time the islands were affected by hydro-isostatic and tectonic changes which resulted in a c. 1.75m drop in sea-level – only stabilising around

<sup>1</sup> The Lapita Cultural Complex stretched from the Bismarck archipelago to Samoa (around 3500 to 2400 years ago) (Green 1979).

1500 years ago (Dickinson 2000, Athens et al. 2004, Nunn and Carson, 2015). The impact of this process is considered to be evident in the decline of certain shell taxa found in early midden deposits (Butler 1995, Amesbury 2007, Carson and Hung 2017).

Clearly, our understanding of the most fundamental characteristics of the initial movement into Remote Oceania is poor and requires re-evaluation. The solution to this dilemma is to establish the exact timing of first settlement and subsequent activities throughout the Mariana Archipelago and beyond. This means our understanding of the  $^{14}\text{C}$  variation of the most common element in these archaeological sites – shell – has to be improved. Moreover, with flood levels predicted to rise by 1.98m by 2070 (<https://coast.noaa.gov/digitalcoast/stories/CNMI-SLR.html>) and reefs in the region now threatened by sedimentation (van Beukering et al. 2006) it is important that we fully understand the impact such changes have on these small island ecosystems.

The isotopic composition of mollusc shells primarily reflects the environment they live in. Shells recovered from archaeological sites, therefore, give us a way to study paleoclimate and paleo-environmental change that is directly correlated to human activity (Prendergast and Stevens 2014). Molluscs are widely distributed across a wide range of environments, occur at all latitudes, and offer greater potential for high-resolution chronometric and geospatial analysis than most other paleo-environmental proxies used today. They are, however, one of the most complex proxies to interpret; in the more restrictive coastal lagoon and estuarine settings, carbon from a range of sources can impact on the shells, resulting in both marine and terrestrial inputs and  $^{14}\text{C}$  ages that appear either too young or too old. Careful selection of shell taxa depending on habitat and diet can help, but species-specific diversity and the ability of some animals to adapt to a variety of environmental conditions leads to problems when interpreting shell dates. One concern is the presence of bicarbonate ions – generated by seepage through calcareous strata – which can become incorporated into the shells of animals living in the water, and result in  $^{14}\text{C}$  ages that are too old. This possibility for Saipan was recognised more than 50 years ago by the radiocarbon team at Chicago. They suggested a 1500-year correction to an oyster shell date (C-669;  $3479 \pm 200$  BP) from the site of Chalan Piao (Cloud et al. 1956:4), but this was never investigated further.

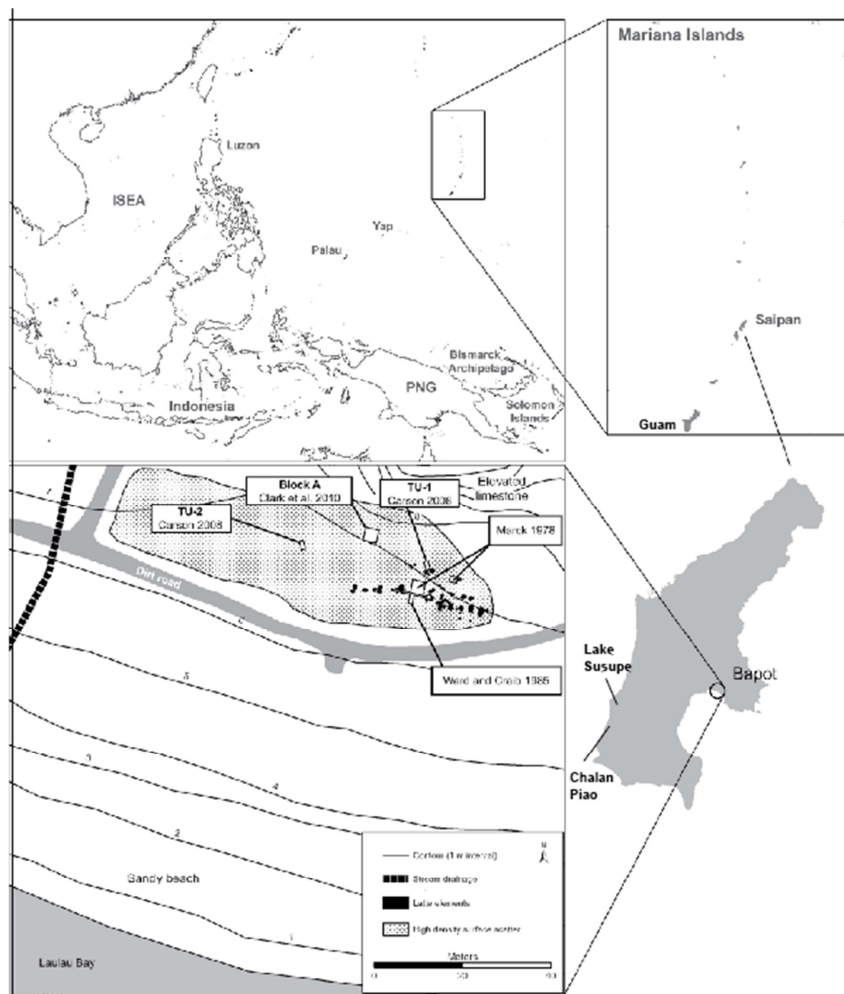


Figure 1. Map of the North-western Pacific showing excavations undertaken at Bapot-1 on Saipan in the Mariana Archipelago.

Petchey and Clark (2011) suggested that it may be possible to predict “hardwater” offsets in shellfish by combining  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  with the  $^{14}\text{C}$  results; in the tropics  $\delta^{18}\text{O}$  records change in salinity (less-saline waters are typically terrestrial in origin), while the  $\delta^{13}\text{C}$  value of marine shells predominantly reflects water source (low productivity terrestrial waters have depleted  $^{13}\text{C}$  values). Moreover, they suggested that isotopic change over time may reflect changing nearshore conditions in regions affected by deforestation, tectonic movement or sea-level change (Petchey et al. 2013:77). Further support for these hypotheses was obtained at the site of Bapot-1, on Saipan in the Mariana Islands, where estuarine *Anadara antiquata* shells from the earliest archaeological deposits had  $^{14}\text{C}$  offsets of up to 300 years, and  $\delta^{13}\text{C}$  values significantly different to reef dwelling animals (Petchey et al. 2017). Petchey et al. (2017:123) also suggested that this hardwater  $^{14}\text{C}$  offset could be variable in response to a drop in sea-level over the last 4000 years as indicated by a change in the archaeological shellfish remains, and the palynological and geological evidence (Butler 1995, Dickinson 2000, Amesbury 2007).

While changing  $^{14}\text{C}$  reservoirs and multiple carbon sources may seem to have obvious problems for our ability to develop precise shell radiocarbon chronologies for archaeological, geological and paleo-environmental research, the reverse may in fact be true. Identification of isotopic anomalies may prove to be immensely useful for documenting environmental change, while sites affected by hardwater should increase our ability to detect  $^{14}\text{C}$  offsets that would normally go undetected when evaluating animals that are in equilibrium with the global marine  $^{14}\text{C}$  reservoir. Moreover, by studying specific taxa that adapt to water conditions, rather than those that have narrow habitat tolerances, we can track change over time. This paper presents the first systematic study of

marine and estuarine  $^{14}\text{C}$  variation for bivalves from Bapot-1, and the first study investigating isotopic response to sea-level changes over the first 1000 years of Mariana settlement.

### 1.1. Mariana Islands: environmental and archaeological evidence.

The site of Bapot-1 is located in Lao Lao Bay on the southeast coast of Saipan, Northern Mariana Islands (Figure 1). To the east of the bay, karst limestone bedrock dominates, while volcanic soils and bedrock are found to the west (Cloud 1959). Most of the available fresh groundwater on Saipan comes from the Mariana Limestone aquifer, the water level of which can fluctuate in response to tide and other changes in sea-level (Carruth 2003). Conventional understanding of reservoir offsets (Stuiver et al. 1986, Petchey et al. 2008) would suggest that water in Lao Lay Bay – well washed by northeast trade wind-generated waves (Houk et al. 2011:8) – should have dissolved inorganic carbon (DIC)  $^{14}\text{C}$  values in equilibrium with the open ocean. However, modern salinity profiles across the bay display a freshwater content that changes in response to tides and rainfall (Houk et al. 2011:4-5).

Saipan has undergone both tectonic and hydro-isostatic sea-level change. Dickinson (2000:737) estimated an average of 1.75m emergence of the island since the mid-Holocene due to hydro-isostatic uplift in the interval between 4750 and 2250 BP. Athens and Ward (2005:53), refined this observation using radiocarbon dates and pollen curves from Lake Susupe, southwest Saipan. They placed the highstand at c. 3,000 BP, followed by rapid sea-level fall after c. 2,500 BP, finally reaching modern heights by about 1,500 years ago. A date of 2455-2298 cal. BP (68% prob.)<sup>2</sup> obtained from sediments on top of coral (the Merizo limestone that formed during the mid-Holocene highstand in Guam), indicated that reef building had declined and coastal infilling had begun (Athens and Ward 2005:26). This lowering sea-level almost certainly impacted on the local aquifers and has been cited as a causal factor behind changing mollusc remains in archaeological sites (Carson and Hung 2017), of note being an early dominance of *Anadara antiquata* followed by *Strombus* species c. AD 1000 (Butler 1995, Graves and Moore 1985; Leidemann 1980). Amesbury (2007) attributed this shift to associated changes in the distribution of mangroves, which would have impacted detrimentally on the *Anadara antiquata* living in these silty habitats.

### 1.2. Shell Isotopes

Most shellfish precipitate their shells in equilibrium with the isotopic signature of DIC from the waters they live in (marine carbonate  $\delta^{13}\text{C} = 0\text{-}2\text{‰}$ ; Gupta and Polach 1985:114). This can however be complicated by “vital effects” (i.e., growth, diet and respiration) and specific habitat characteristics (McConnaughey et al. 1997; Lorrain et al. 2004). In particular, a small amount of carbon in shells (<10%)<sup>3</sup> is dietary in origin (McConnaughey et al. 1997, McConnaughey and Gillikin 2008). For most studies, these effects are assumed to have little significant impact on the  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  since DIC dominates and dietary sources of carbon are typically in equilibrium with the consumer’s environment. It is, therefore, assumed that input of freshwater within an ocean environment should result in the depletion of shell  $^{13}\text{C}$  and  $^{18}\text{O}$  (Swart et al. 1983; Gat 1996:241, 255; Goewert et al. 2007; McConnaughey and Gillikin 2008), while increased productivity and  $\text{CO}_2$  atmospheric absorption in reef/intertidal locations may result in enrichment in  $^{13}\text{C}$  (Weber and Woodhead 1971; Watanabe et al. 2006).

The impact on  $^{14}\text{C}$ , however, is potentially more dramatic. The surface ocean DIC (down to around 200 m depth) has an apparent  $^{14}\text{C}$  age that is, on average, 410 years older than associated terrestrial materials (Stuiver et al. 1986). Consequently, in estuarine environments the introduction of terrestrial sources of DIC or particulate carbon from the absorption of atmospheric  $\text{CO}_2$ , or the incorporation of freshwater from rivers, typically results in small shifts towards younger ages (c. -40 years for 10% contribution from “modern”  $^{14}\text{C}$ ) (Stuiver and Braziunas 1993; Southon et al. 2002; Guilderson et al. 2000). This shift ( $\Delta\text{R}$ ) from the global marine reservoir is often undetectable in archaeological chronologies. Conversely, 10% “dead”  $^{14}\text{C}$  added to a modern shell will increase the age by c. 770 years. Old ages, caused by hardwater or the upwelling of  $^{14}\text{C}$ -depleted water, is a long-recognised area of concern (Stuiver and Braziunas 1993, Dye 1994, Ingram and Southon 1996,

<sup>2</sup> This date includes a regional reservoir correction ( $\Delta\text{R}$ ) of  $115 \pm 50$   $^{14}\text{C}$  years. Coral debris infilling the coral gave a conventional radiocarbon age (Beta-129463) of  $2800 \pm 60$   $^{14}\text{C}$  years BP (Athens and Ward 1999:141). Using the  $\Delta\text{R}$  of  $-55 \pm 27$   $^{14}\text{C}$  years calculated for marine DIC from the Bapot-1 research, Beta-129463 now calibrates to 2760-2400 cal. BP (95% prob.).

<sup>3</sup> Considerably uncertainty in this value exists. McConnaughey and Gillikin (2008:292) note that estimates range up to 90%.

Spenneman and Head 1998, Anderson et al. 2001, Culleton et al. 2006, Petchey and Ulm 2010, Petchey and Clark 2011, Holmquist et al. 2015). Less well-studied is the increase mobilization of aged carbon caused by human disturbance (Butman et al. 2014).

Old shell  $^{14}\text{C}$  ages can be caused by a multitude of factors, including the incorporation of older “natural” shell deposits, disturbance of layers and upwelling of ancient marine waters. In the Pacific c. 25 percent of islands are limestone or a limestone composite (Nunn et al. 2016). However, the exact magnitude of error introduced by hardwater is considered to be dependent on the rate of water exchange with the open ocean (the “residence time”) and therefore on current flow, the presence of bays and lagoons, freshwater hydrology and geology (Gómez et al. 2008, Petchey et al. 2008) and cannot be predicted by the presence of limestone alone. Moreover, the impact on shellfish depends on the habitat and dietary preferences of specific species and their tolerances for low salinity waters (Ascough et al. 2005, Petchey et al. 2012, 2013, Holmquist et al. 2015, Lindauer et al. 2017). Estuarine shellfish are more likely to be influenced by hardwater because of their preference to inhabit sheltered bays with reduced water exchange with the open ocean, but many mollusc species are tolerant of a range of water conditions (Reimer 2014, Hogg et al. 1998) and it can be difficult to determine what samples to date and what  $\Delta\text{R}$  is appropriate.

In environments where extraneous influences on  $^{14}\text{C}$  are possible, shell  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  in combination with  $^{14}\text{C}$  can help identify the cause of this offset. This multiproxy approach to age determination is especially important when evaluating hardwater impact because  $\delta^{13}\text{C}$  approximates that of the source rocks (i.e.,  $\delta^{13}\text{C} = 0\text{‰}$ ) and is therefore masked, but decreased salinity and negative  $\delta^{13}\text{C}$  values can be used to identify any non-marine influence on shellfish and, with older than expected  $^{14}\text{C}$  ages, confirm a hardwater effect. Using a range of proxy data, short-term localised changes to  $^{14}\text{C}$  have been documented in estuaries due to anthropogenic interference (Sabatier et al. 2010), rainfall (Culleton et al. 2006, Philippsen et al. 2013), changes to groundwater drainage and land usage (Gómez et al. 2008), upwelling (Petchey et al. 2008b), and to infer hardwater input (Petchey and Clark 2011).

Longer term shifts in marine  $^{14}\text{C}$  between 4000- and 1900-years BP have been linked to changes in ocean circulation (Yu et al. 2010, Hua et al. 2015, Komugabe-Dixson et al. 2016), but are poorly studied over the period of human settlement of the Pacific. Consequently, modern validation studies and reservoir correction values (Hogg et al. 1998, Cook et al. 2004, Petchey and Clark 2010) may not be applicable to archaeological material. Unfortunately, the use of archaeological “paired” terrestrial (charcoal) and marine samples to test potential marine offsets (e.g., Clark et al. 2010, Carson 2010, 2017) and Bayesian approaches to  $\Delta\text{R}$  calculation (Macario et al. 2015) are complicated by site disturbance, poorly consolidated sandy deposits and beach slopes with areal spread, variation in hydrology and coastline morphology (Petchey et al. 2017).

## 2. Methodology

In complicated situations where  $\Delta\text{R}$  is likely to change regionally, over time and by taxa, a new methodology for dating sites is essential. Here we investigate the potential of a two-tiered approach to dating the site of Bapot-1. The first section investigates stable isotope variation in shell over time. The second involves selection of samples for dating based on the hypothesis that  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  can help detect estuarine versus marine influence on shells, and be used to establish a marine and estuarine  $\Delta\text{R}$  offsets for the site.

### 2.1. Sample selection

Shells for  $\delta^{13}\text{C}$ ,  $\delta^{18}\text{O}$  and  $^{14}\text{C}$  analysis were obtained from Unit 4, Block A excavations undertaken in 2008 (Figure 2). Radiocarbon ages from this unit range from 510 BP to 3810 BP (Clark et al. 2010). Shells were selected from archived samples excavated by 10cm spits that followed the deposition layers; Layer I (Cat #22; depth 48/50-55 cm), Layer III (Cat #40, 49; depth 80-100 cm), Layer IV (Cat #67, 76, 84; depth 110-140 cm), Layer V (Cat #100, 108, 124, 132, 141; depth 150-210 cm), Layer VI (Cat #149; depth 210-220 cm) and Layer VII (Cat #163, 166, 169, 174; depth 230-260 cm). No samples from Layer II were analysed. The mollusc remains are discussed by O’Day (2015). The species selected include: *Gafrarium pectinatum*, *Anadara antiquata*, *Quidnipagus* sp. and

*Tridacna* sp. [bivalves]; *Trochus* sp. (probably Tegulidae family), *Turbo* sp., *Monetaria monetaria*<sup>4</sup>, and *Canarium mutabilis*<sup>5</sup> [gastropods].

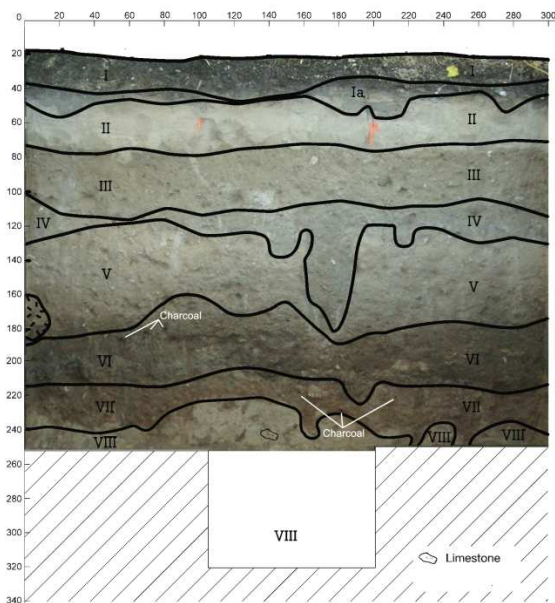


Figure 2: Stratigraphy of Block A, Bapot-1, North profile (Unit 1–Unit 3).

Several of these taxa are considered problematic for  $^{14}\text{C}$  analysis especially in limestone locations because of the ingestion of carbonate, either directly or indirectly via algal grazing. These include *Quidnipagus* sp. (*Tellina*) (Dye 1994, Hogg et al. 1998), *Cypraea* sp. (Dye 1994), *Strombus* (Petchey et al. 2012, 2013), *Trochus* and *Turbo* spp. (Petchey et al. 2015). The interpretation of  $^{14}\text{C}$  results obtained from these algal grazing and deposit-feeding shellfish is complex; ingesting c. 10% ancient carbonate could impact on their age even though their shell carbonate primarily comes from the marine waters they live in. Thus, their shell isotopes would appear marine but the shell could still be influenced by old sources of carbon. Although terrigenous sediment found in Lao Lao Bay is derived from volcanic sources (Randall 1991), limestone rocks are exposed on the coast less than 1km from Bapot-1. We have included these taxa in the  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  analyses for comparison purposes, but do not consider here any  $^{14}\text{C}$  results on these animals.

*Gafrarium* and *Anadara* occupy similar environmental niches. *Gafrarium* spp. are preferentially found in inner-lagoon and high intertidal regions within seagrass beds and mangrove forests (Baron and Clavier 1992; Tebano and Paulay 2000:9–10). *A. antiquata* are found in waters that are regularly exposed and submerged by tides. They also prefer less saline estuarine waters, but the taxa as a whole occupy niches in many different environments (Broom 1985:6-7). However, as individual species they do not tolerate change and quickly die once the environment changes (Davenport and Wong 1986). *Gafrarium* is much more tolerant of changing environmental conditions and has a lower salinity tolerance than *Anadara* (Davenport and Wong 1986, McMahon 2003:488). Tridacninae are found in reef locations and prefer full strength, clear seawater and will quickly die if exposed to brackish or freshwater for long periods (Ellis 1999, Hart et al. 1998). Petchey and Clark (2011) theorised that adult Tridacninae may have elevated (i.e., younger)  $^{14}\text{C}$  values because in addition to filter-feeding they also obtain energy from photosynthetically derived carbohydrate via a symbiotic relationship with zooxanthellae. Unfortunately, we were not able to identify species in the Bapot-1 specimens, but all are small (<10cm) and are probably juveniles.

To obtain a representative indication of isotopic spread across the estuarine and reef/marine environment, five shells of each taxa were sampled from every second spit for  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  analysis.

<sup>4</sup> Also known as *Cypraea monetaria*.

<sup>5</sup> Also known as *Strombus mutabilis mutibilis*.



After stable isotope evaluation, specific shell samples were selected for  $^{14}\text{C}$  based on a prediction of estuarine or marine origin. Twenty-nine shell samples were taken for  $^{14}\text{C}$  analysis from throughout Unit 4 (layers III, IV, V and VI). Clark et al. (2010) and Petchey et al. (2017) have already reported a number of  $^{14}\text{C}$  dates from Layer VII of Unit 4 (230-260cm), a further 2 charcoal dates were also obtained for comparison from Layer V (180-190cm) and Layer IV (130-140cm). This brings the total number of dates from Unit 4 to 42. The total includes short-lived charcoal (n=2), charcoal of unknown species (n=3), bird bone (n=1), *Anadara* (n=17), *Gafrarium* (n=13), *Tridacna* (n=5) and a *Conus* ring artefact (n=1).

## 2.2. Pretreatment

Bivalve shell is grown by successive addition of calcium carbonate from the umbo (hinge) to the ventral (lip) margin. Gastropod growth starts at the apex with the youngest material at the shell lip and several years of growth is concentrated in the callus and columella<sup>6</sup> providing well-averaged samples (Culleton et al. 2006:396). Ideally, dating the last few growth rings of bivalves or the callus/columella of gastropods will provide the  $^{14}\text{C}$  age at death. Where possible a sample c. 10 mm-long and 4 mm-wide was taken parallel to the margin/lip of each shell using a Dremel® 3000 Rotary Tool fitted with a diamond wheel. This selection process is designed to avoid seasonal variation and give an average value comparable to the decadal resolution of the  $^{14}\text{C}$  calibration curves (Culleton et al. 2006, Petchey et al. 2008). Many of the gastropod samples from Bapot-1 were fragmentary and it was not possible to follow this sampling protocol in all instances. All shells sampled are naturally deposited as aragonite – avoiding isotopic differences between aragonite and calcite.<sup>7</sup>

## 2.3. Stable Isotope Analysis.

$\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values were measured at the University of Waikato using a cavity ring-down  $\text{CO}_2$  isotope analyser (CRDS) (Los Gatos Research model CCIA-46). Phosphoric acid (102%) was added to each ground shell sample (0.42-0.5 mg) to evolve  $\text{CO}_2$ . Samples were heated ( $72^\circ\text{C}$ ,  $\geq 1$  hr) to promote hydrolysis before stable isotope analysis. Pressure corrections were made using an in-house standard of ground pipi shell (*Paphies australis*). IAEA (International Atomic Energy Agency) standards NBS-18 (calcite;  $\delta^{13}\text{C} = -5.014\text{‰}$ ,  $\delta^{18}\text{O} = -23.2\text{‰}$ ) and NBS-19 (limestone;  $\delta^{13}\text{C} = 1.95\text{‰}$ ,  $\delta^{18}\text{O} = -2.20\text{‰}$ ) were used to construct a two-point isotope calibration curve and further evaluated using BDH ( $\delta^{13}\text{C} = -24.95\text{‰}$ ,  $\delta^{18}\text{O} = -13.99\text{‰}$ ) and Sigma ( $\delta^{13}\text{C} = -14.18\text{‰}$ ,  $\delta^{18}\text{O} = -20.07\text{‰}$ ) synthetic  $\text{CaCO}_3$  standards (Beinlich et al. 2017, Table 2). A drift correction was made after every two samples using 1500 ppm  $\text{CO}_2$  reference gas.  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values are reported as ‰ V-PDB, and the standard deviation of 0.4‰ was determined using sample reproducibility of duplicate measurements. Where possible all  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  results previously reported in Clark et al. (2010) and Petchey et al. (2017) run by IRMS (Isotope Ratio Mass Spectrometry) were re-measured by CRDS to ensure consistency.

## 2.4. Radiocarbon Dates

Samples for  $^{14}\text{C}$  were prepared in the AMS facility at the Radiocarbon Dating Laboratory, University of Waikato. Shell (< 3 mm fragments, 35–45 mg) were etched in 0.1M HCl at  $80^\circ\text{C}$  to remove c. 45% of the surface (Burr et al. 1992), and then tested for recrystallization by Feigl staining (Friedman 1959) to make sure only aragonite was present in the shell.  $\text{CO}_2$  was collected from shells by reaction with 85%  $\text{H}_3\text{PO}_4$  and cryogenically separated  $\text{CO}_2$  was reduced to graphite with  $\text{H}_2$  at  $550^\circ\text{C}$  using an iron catalyst. Pressed graphite was analysed at the Keck Radiocarbon Dating Laboratory, University of California (Southon and Santos [2007] and references therein). Six primary OxII standards were used to set up and tune the AMS system as well as to normalize the  $^{14}\text{C}/^{12}\text{C}$  ratios (c.f., Santos et al. 2007). One blank [in-house Carrara marble blank (Fm = 0.002)] and an in-house shell (*Tridacna*) standard (Fm = 0.686; c. 3028-year BP) were used for background correction and

<sup>6</sup> Callus is the thick, shiny secondary deposit of shell around the opening. Columella is the central coiling axis of the shell.

<sup>7</sup> A difference in  $\delta^{18}\text{O}$  between calcite and aragonite occurs in some shellfish that deposit  $\text{CaCO}_3$  in both forms (Rick et al. 2006). This has been attributed to differential equilibrium conditions between the interior and exterior of the shell rather than to shell chemistry (Keith et al. 1964).

quality control. All  $^{14}\text{C}$  results were fractionation-corrected using the online AMS  $\delta^{13}\text{C}$  values which are not reported.

### 3. Results

259 shells were sampled for  $^{18}\text{O}$  and  $^{13}\text{C}$ . Results are summarised in Table 1 and Figure 3 with individual values given in Supplementary Tables 1 and 2. Radiocarbon dates from Unit 4 are given in Supplementary Table 3 and summarised in Figure 5. All radiocarbon dates were calibrated in OxCal v4.3 (Bronk Ramsey 1995) using the Marine13 and Intcal13 calibration curves (Reimer et al. 2013).

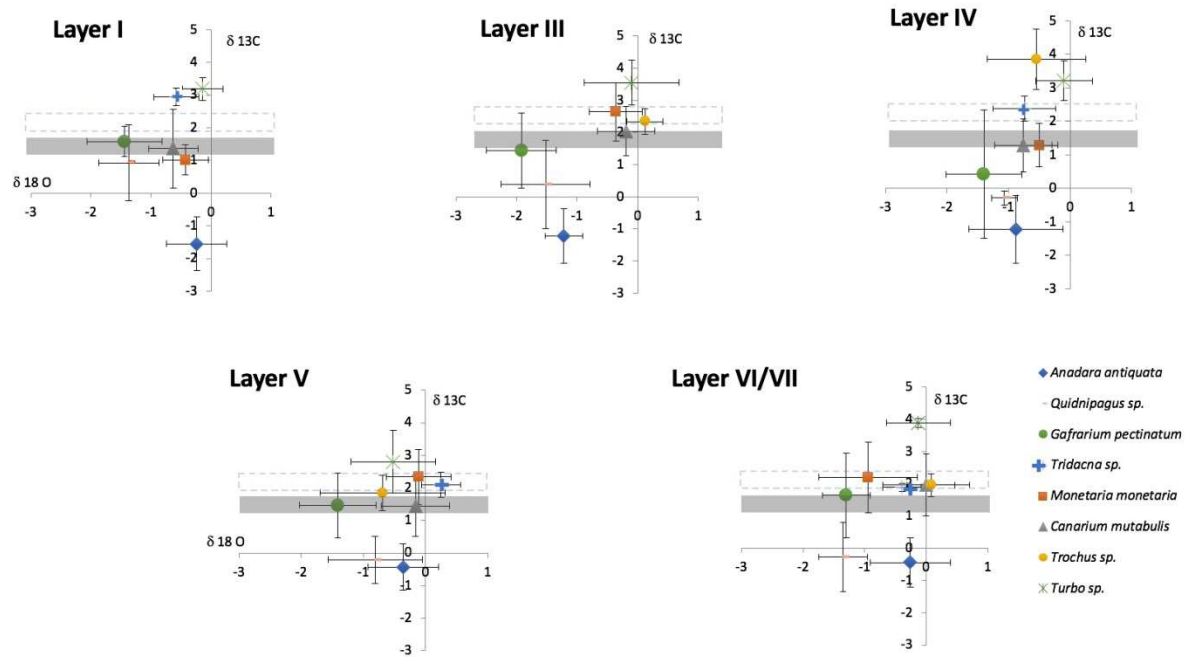


Figure 3. Average and standard deviation of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values of shellfish from Bapot-1 by layer. The grey bar marks the modelled  $\delta^{13}\text{C}$  of the modern surface ocean DIC around Saipan (between 1.3 and 1.7‰) after Tagliabue and Bopp (2008, Fig. 2). The dashed box approximates the predicted  $\delta^{13}\text{C}$  for surface ocean DIC prior to the 19<sup>th</sup> century (based on data from Böhm et al. 1996).

Layer	I	II	III	IV	V	VI	VII
Species	<i>Anadara antiquata</i>						
Average $\delta^{13}\text{C}$ (‰)	-1.55	-	-1.22	-1.22	-0.43	-0.47	-0.46
$\delta^{13}\text{C}$ Standard Deviation	0.82	-	0.85	1.01	0.71	0.73	0.88
Average $\delta^{18}\text{O}$ (‰)	-0.24	-	-1.22	-0.88	-0.36	0.07	-0.35
$\delta^{18}\text{O}$ Standard Deviation	0.50	-	0.31	0.77	0.57	0.88	0.63
Number of Samples	2*	-	7	10	10	5	23***
Species	<i>Gafrarium pectinatum</i>						
Average $\delta^{13}\text{C}$ (‰)	1.58	-	1.44	0.42	1.46	2.27	1.00
$\delta^{13}\text{C}$ Standard Deviation	0.47	-	1.17	1.91	0.10	0.78	1.46
Average $\delta^{18}\text{O}$ (‰)	-1.44	-	-1.92	-1.41	-1.41	-1.30	-1.31
$\delta^{18}\text{O}$ Standard Deviation	0.62	-	0.58	0.61	0.62	0.29	0.48
Number of Samples	5	-	10	10	10	5	5
Species	<i>Quidnipagus palatam</i>						
Average $\delta^{13}\text{C}$ (‰)	0.93	-	0.39	-0.29	-0.21	0.07	-0.61
$\delta^{13}\text{C}$ Standard Deviation	1.15	-	1.37	0.21	0.73	1.40	0.51
Average $\delta^{18}\text{O}$ (‰)	-1.37	-	-1.52	-1.07	-0.81	-1.50	-1.20
$\delta^{18}\text{O}$ Standard Deviation	0.50	-	0.74	0.21	0.76	0.41	0.33
Number of Samples	5	-	5	2	5	5	5
Species	<i>Tridacna sp.</i>						
Average $\delta^{13}\text{C}$ (‰)	2.94	-	-	2.38	2.09	-	1.89
$\delta^{13}\text{C}$ Standard Deviation	0.27	-	-	0.37	0.38	-	0.13
Average $\delta^{18}\text{O}$ (‰)	-0.58	-	-	-0.74	0.25	-	-0.39
$\delta^{18}\text{O}$ Standard Deviation	0.37	-	-	0.51	0.31	-	0.31
Number of Samples	2	-	**	3	1	-	1
Species	<i>Monetaria monetaria</i>						
Average $\delta^{13}\text{C}$ (‰)	1.01	-	2.65	1.29	2.33	-	2.20
$\delta^{13}\text{C}$ Standard Deviation	0.46	-	0.90	0.64	0.90	-	1.10
Average $\delta^{18}\text{O}$ (‰)	-0.43	-	-0.36	-0.50	-0.11	-	-0.94

$\delta^{18}\text{O}$ Standard Deviation	0.38	-	0.44	0.30	0.52	-	0.80
Number of Samples	1	-	4	4	10	-	10
Species	<i>Canarium mutabilis</i>						
Average $\delta^{13}\text{C}$ (‰)	1.36	-	2.04	1.29	1.44	-	1.97
$\delta^{13}\text{C}$ Standard Deviation	1.20	-	0.76	0.79	0.94	-	0.95
Average $\delta^{18}\text{O}$ (‰)	-0.63	-	-0.19	-0.76	-0.16	-	0.0
$\delta^{18}\text{O}$ Standard Deviation	0.41	-	0.47	0.47	0.55	-	0.70
Number of Samples	5	-	6	10	14	-	10
Species	<i>Trochus sp.</i>						
Average $\delta^{13}\text{C}$ (‰)	-	-	2.34	3.86	1.85	-	1.95
$\delta^{13}\text{C}$ Standard Deviation	-	-	0.41	0.90	0.54	-	0.36
Average $\delta^{18}\text{O}$ (‰)	-	-	0.12	-0.55	-0.69	-	0.08
$\delta^{18}\text{O}$ Standard Deviation	-	-	0.30	0.80	1.01	-	0.39
Number of Samples	-	-	1	3	4	-	1
Species	<i>Turbo sp.</i>						
Average $\delta^{13}\text{C}$ (‰)	3.19	-	3.55	3.21	2.80	-	3.89
$\delta^{13}\text{C}$ Standard Deviation	0.35	-	0.70	0.59	0.96	-	0.49
Average $\delta^{18}\text{O}$ (‰)	-0.14	-	-0.10	-0.10	-0.52	-	-0.13
$\delta^{18}\text{O}$ Standard Deviation	0.34	-	0.78	0.46	0.68	-	0.52
Number of Samples	5	-	9	10	13	-	3

**Table 1:** Average  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  and associated standard deviations for each shell taxa recovered from layers I to VII.

\*  $^{14}\text{C}$  dates on both samples indicate they do not originate from this layer and probably come from layers V to VII.

\*\* *Tridacna* was found in this layer but none were available for analysis.

\*\*\* Additional *Anadara* shells were sampled from Layer VII to try and identify shells with higher  $\delta^{13}\text{C}$  values.

Disturbance in the top c. 1.5m of the site (layers IV and above) presented difficulties when evaluating change in  $^{18}\text{O}$  and  $^{13}\text{C}$  over time. However, pockets of indurated sand, first encountered in Layer III and increasing in induration downwards through VI and VII, would have limited mixing between earlier and later activities. This is supported by ceramic finds within these layers (Winter 2015); thin red-slipped pottery from carinated jars, including some “Achuago Incised” and “San Roque” sherds, were found in layers V, VI and VII.<sup>8</sup> Thick-walled, red-slipped ceramics and grey thick-walled ceramics from flat based trays start to appear in the upper levels of Layer IV (c. 150cm) (Winter 2015:174, Figure 59). Increasing quantities of thick-walled/red ceramics start to appear in layers III and II, but only a handful of early thinware ceramics were recovered (Winter 2015; Table 3) indicating minor upward displacement of this early material. We, therefore, conclude that these lower layers are uncontaminated by younger material and can be used to evaluate hardwater and species-specific offsets.

Shellfish taxa also change over time. *Anadara antiquata* numbers drop significantly from Layer III (100cm) upwards, and are rare by Layer I (2 values in Layer I [4.2% of shells in layer], 25 valves [18%] in Layer III, 109 valves [12%] in Layer IV, 521 valves [27%] in Layer V; 185 valves [26%] in Layer VI, and 108 valves [21%] in Layer VII) (O’Day 2015). Throughout the sequence, the number of individual specimens (NSIP) of gastropods and bivalves tend to show a similar trend with a trough at c. 170-190cm (lower spits of Layer IV). A change was also found in the upper levels of Layer IV (110-140cm) where bivalves – primarily *Gafrarium* spp. – become dominant (Figure 4), while shellfish numbers generally drop by half (O’Day 2015:182). The possibility that a high proportion of the shell material in the upper layers (layers III to I) could have been re-deposited from the earlier layers, was not apparent until  $^{14}\text{C}$  dates were obtained. Consequently, the following evaluation of shellfish isotopes and magnitude of hardwater offset concentrates on layers V to VII.

<sup>8</sup> San Roque decoration consists of stamped circles. Achuago ceramics may be slipped in red, black or buff; decoration consists of parallel incised lines in rectilinear or curved patterns around the neck of the pot with spaces filled with stamped circles or punctuations. Both styles had lime filled decoration and similar vessel shapes – round bottoms with small carination’s and everted rims. The relationship between both styles is unclear, but Rainbird (2004:82-83) considered it likely that San Roque ceramics were younger because the decorative elements continue into the later ceramic period.

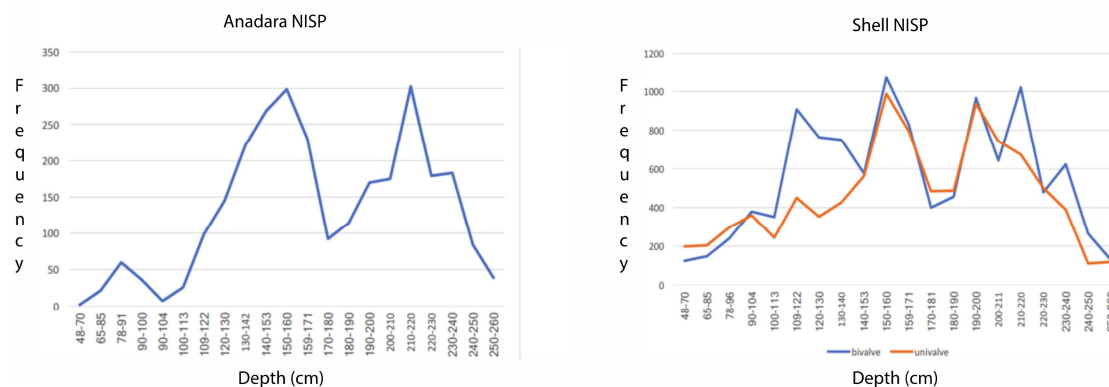


Figure 4: Shellfish number of individual specimens (NISP) for all units, Bapot-1. A) Change over time in *Anadara* shells. B) Change over time in bivalves relative to gastropods (univalves).

### 3.1. Layers V to VII

The gastropods (*Canarium mutabilis*, *Trochus* sp., *Turbo* sp., and *Monetaria monetaria*) are found in sandy and rocky shore sub-tidal and reef slope environments, and are herbivorous animals that could potentially ingest sediment (Poutiers 1998, Dumas et al. 2017). The  $\delta^{13}\text{C}$  values of *Turbo* and *M. monetaria* shells are enriched by 1-2‰ relative to ocean water values ( $3.99 \pm 0.96\text{‰}$  and  $2.27 \pm 0.95\text{‰}$  respectively; see Figure 3), in keeping with reported enrichment for shells from lagoons with high productivity (Romanek et al. 1992). *Trochus* and *C. mutabilis* have lower average  $\delta^{13}\text{C}$  values ( $1.87 \pm 0.37\text{‰}$  and  $1.63 \pm 0.92\text{‰}$  respectively). There is no obvious change between layers V to VII, though  $\delta^{13}\text{C}$  values for all the gastropod taxa tend to be more variable than the bivalves (Table 1) and may reflect diet, seasonality, daily changes associated with phytoplankton bloom and decay, habitat preferences and/or possible age-related preferences (adults versus juveniles). The gastropod  $\delta^{18}\text{O}$  values cluster around  $-0.39\text{‰}$  indicating a consistent and more saline habitat temperature for these animals.

The *Tridacna* spp. results (average =  $1.91 \pm 0.14\text{‰}$ ) are also higher than the ocean water  $\delta^{13}\text{C}$ . Juvenile *Tridacna* get most of their metabolic carbon from filter feeding, so it is unlikely that there was any significant influence from atmospheric  $\text{CO}_2$  as is possible in larger clams.  $\delta^{18}\text{O}$  values are also uniform (average =  $-0.07 \pm 0.45\text{‰}$ ) and support growth in a marine habitat.

Overall, the  $\delta^{18}\text{O}$  values for *A. antiquata* average  $-0.30 \pm 0.56\text{‰}$  (ranging from  $-1.67\text{‰}$  to  $0.94\text{‰}$ ); similar to the reef shellfish. *Anadara* are the most  $^{13}\text{C}$  depleted of all shellfish studied (average =  $-0.45 \pm 0.75\text{‰}$ ; ranging from  $-1.45$  to  $1.61\text{‰}$ ). This may be explained by their tendency to favour mangrove locations where increased  $^{12}\text{C}$  from the decay of organic matter could have resulted in more negative  $\delta^{13}\text{C}$  values; only one individual out of 38 had a  $\delta^{13}\text{C}$  equivalent to the average ocean value ( $1.3$ - $1.7\text{‰}$ ) for this location (Wk-45616 from Layer VII;  $\delta^{13}\text{C} = 1.61 \pm 3.5\text{‰}$ ,  $\delta^{18}\text{O} = -0.11 \pm 4.0\text{‰}$ ).

The isotope composition of *Gafrarium pectinatum* is very different to the other bivalves. The  $\delta^{18}\text{O}$  values (average =  $-1.35 \pm 0.42\text{‰}$ ) are indicative of less saline waters. The  $\delta^{13}\text{C}$  value ranged from  $-0.99\text{‰}$  to  $2.72\text{‰}$  (average =  $1.55 \pm 1.13\text{‰}$ ), with 11 out of 20 shells sampled having a  $\delta^{13}\text{C}$  above  $1.7\text{‰}$ . *Quidnipagus* have similar  $\delta^{18}\text{O}$  values (average =  $-1.17 \pm 57\text{‰}$ ) to *Gafrarium* but lower  $\delta^{13}\text{C}$  (average =  $-0.25 \pm 86\text{‰}$ ; ranging from  $-1.45\text{‰}$  to  $2.31\text{‰}$ ) relative to the average ocean value, with only one value above  $1.3\text{‰}$ . These taxa are found in coarse sandy environs (Poutiers 1998), and have deposit-feeding behaviours which may be responsible for the observed  $\delta^{13}\text{C}$ .

Using the average ocean  $\delta^{13}\text{C}$  value of  $1.3$ - $1.7\text{‰}$  (after Tagliabue and Bopp 2008) as a divider between “estuarine” and “marine” water DIC values, we selected for dating two “estuarine” *G. pectinatum* (Wk-45922,  $\delta^{13}\text{C} = -0.99\text{‰}$ ; Wk-45904,  $\delta^{13}\text{C} = 0.97\text{‰}$ ) and two “marine” *G. pectinatum* valves (Wk-45919,  $\delta^{13}\text{C} = 2.23\text{‰}$ ; Wk-45903,  $\delta^{13}\text{C} = 2.09\text{‰}$ ) and one indeterminate (Wk-45888,  $\delta^{13}\text{C} = 1.47\text{‰}$ ) *G. pectinatum* valve. Six *A. antiquata* shells have previously been dated from these lower layers (Clark et al. 2010, Petchey et al. 2017) and all have  $\delta^{13}\text{C}$  values below the ocean average and are considered here to be “estuarine” (see Table 1 and Figure 5). We obtained a further three *A.*

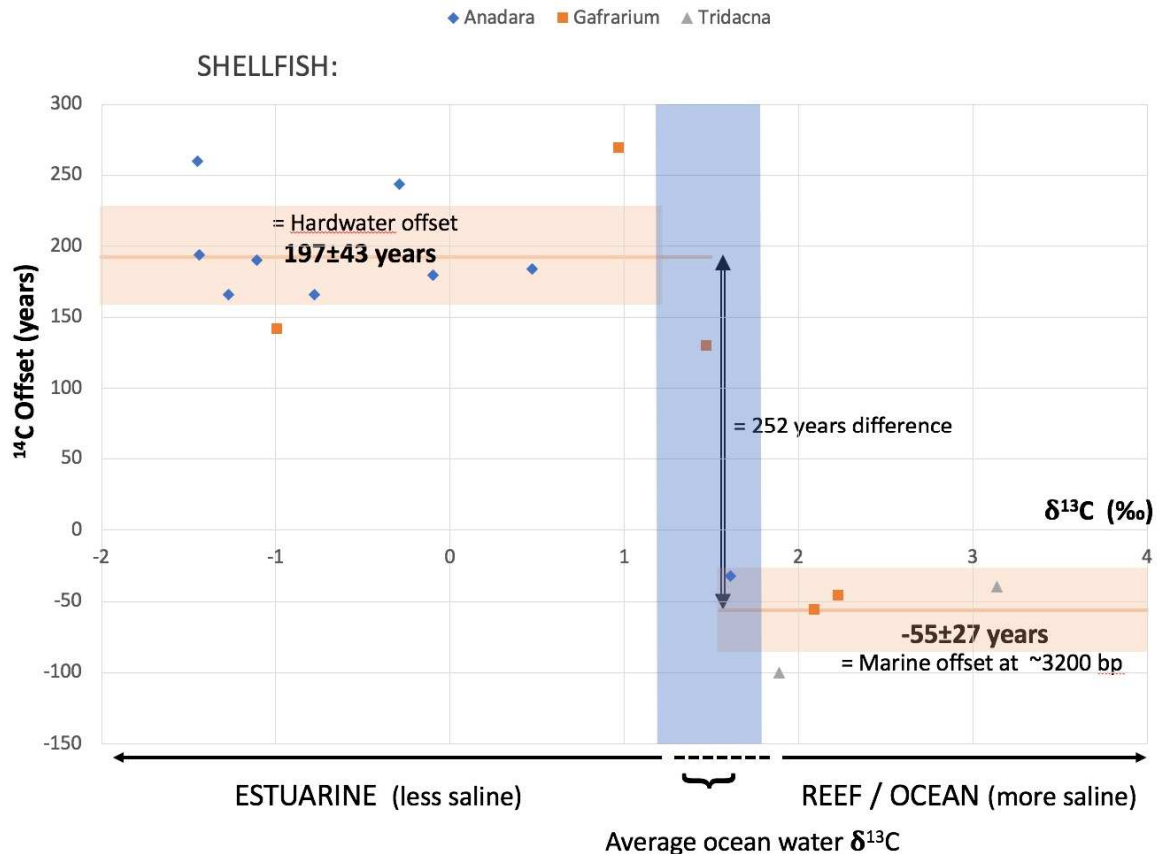
*antiquata*  $^{14}\text{C}$  dates with  $\delta^{13}\text{C}$  values ranging from the lowest (-1.45‰, Wk-45617), to the highest available (1.61‰, Wk-45616). Two *Tridacna* shells considered to represent marine water conditions were also dated; Wk-45892 from Layer V ( $\delta^{13}\text{C} = 2.09\text{‰}$ ) and Wk-45928 from Layer VI ( $\delta^{13}\text{C} = 1.89\text{‰}$ ). A previously dated *Conus* sp. ring (Wk-23771) has a  $\delta^{13}\text{C}$  value of 0.57‰ but is excluded from this comparison. This selected division between marine and estuarine DIC may be too low given the likely seawater-shell (aragonite) enrichment for shells (Romanek et al. 1992) and a decline (<1‰) in modern marine surface  $\delta^{13}\text{C}$  DIC caused by the burning of fossil fuels in the 19<sup>th</sup> century onwards (the Suess effect) (Böhm et al. 1996).

Three charcoal dates are available from Unit 4, layers V to VII; Wk-23768, SANU-55717 and SANU-11619. All three are statistically indistinguishable ( $2958 \pm 18$  BP;  $\chi^2_{2:0.05} = 4.31 < 5.99$ ; GSD=44.75), but only SANU-11619 is a date on a short-lived nut suitable for reservoir age comparison. Using this nutshell age of  $2985 \pm 30$  BP, a  $\Delta\text{R}$  for each shell species was calculated (Table 2). Figure 5 shows the  $\Delta\text{R}$   $^{14}\text{C}$  values for all three shellfish species plotted against  $\delta^{13}\text{C}$ . The two “marine” *Gafrarium* (Wk-45919 and Wk-45903) have  $\Delta\text{R}$  values in-keeping with our marine hypothesis as do the *Tridacna* shells and *Anadara* shell; Wk-45616. Combined, these marine samples have a  $\Delta\text{R}$  of  $-55 \pm 27$   $^{14}\text{C}$  years, which is equivalent to  $\Delta\text{R}$  values obtained from corals for the period between 2500 and 3000 BP for the eastern Australian coastline (average 21 values =  $-84 \pm 69$   $^{14}\text{C}$  years) (Hua et al. 2015, Komugabe-Dixson et al. 2016).<sup>9</sup> Using the  $\delta^{13}\text{C}$  division we calculate an average “estuarine” value of  $197 \pm 43$   $^{14}\text{C}$  years. This suggests a c. 250-year difference between estuarine and open ocean (marine) shells. When these shellfish were alive the input of ancient DIC into the Lao Lao Bay would have had to be less than 5% to shift the ages by this much (5% addition of “dead” carbon would shift the age by c. 390 years).

Shellfish	$\Delta\text{R}$ (yrs)	Chi squared statistics <sup>b</sup>	Environmental division	Environmental $\Delta\text{R}$
<i>Anadara</i> “estuarine” $^{13}\text{C}$ ”	203±15	$\chi^2_{9:0.05} = 14.46 < 16.92$ ; GSD = 31.83	Estuarine	$\Delta\text{R}=197 \pm 12$ ; $\chi^2_{12:0.05} = 12.16 < 21.03$ ; GSD = 42.83
<i>Gafrarium</i> “estuarine” $^{13}\text{C}$ ”	181±24	$\chi^2_{2:0.05} = 7.02 < 5.99$ ; GSD = 77.02		
<i>Gafrarium</i> “marine $^{13}\text{C}$ ”	-51±29	$\chi^2_{1:0.05} = 0.03 < 3.84$ ; GSD = 7.07	Marine	$\Delta\text{R}=-55 \pm 18$ ; $\chi^2_{4:0.05} = 1.75 < 9.49$ ; GSD = 26.74
<i>Anadara</i> “marine $^{13}\text{C}$ ”	-32±42	-		
<i>Tridacna</i>	-70±28	$\chi^2_{1:0.05} = 1.13 < 3.84$ ; GSD = 42.43		

Table 2: Average  $\Delta\text{R}$  for each shellfish taxa studied from Bapot-1, Unit 4, layers VI to VII.  $\Delta\text{R}$  calculated using <http://calib.org/deltar/> (Reimer and Reimer 2017).

<sup>9</sup> Three  $\Delta\text{R}$  values on pre-AD 1950 shells are recorded for the Mariana Islands (Petchev and Clark 2010), but all are gastropods and are questionable due to limited documentation and possible ingestion of limestone and are, therefore, considered unsuitable for calculating a reservoir offset at Bapot-1.



**Figure 5.**  $\delta^{13}\text{C}$  versus  $^{14}\text{C}$  offset ( $\Delta R$ ) showing isotopic separation between estuarine and marine shellfish from Bapot-1 (layers V to VII)

### 3.2. Layer IV

A similar isotope pattern for the reef gastropods is found in Layer IV (Table 1 and Figure 3). *Turbo* and *Trochus* have higher  $\delta^{13}\text{C}$  values (average =  $3.21 \pm 0.56\text{‰}$  and  $3.86 \pm 0.73\text{‰}$ ). *M. monetaria* and *C. mutabilis*  $\delta^{13}\text{C}$  values are, however, low (average =  $1.28 \pm 0.65\text{‰}$  and  $1.29 \pm 0.77\text{‰}$ ) compared to the average ocean value. *Tridacna* results are similarly elevated relative to ocean water  $\delta^{13}\text{C}$  with comparable values to the earlier layers (average  $\delta^{13}\text{C} = 2.38 \pm 0.37\text{‰}$ ; average  $\delta^{18}\text{O} = -0.74 \pm 0.52\text{‰}$ ).

The *G. pectinatum* shells from Layer IV are more depleted in  $^{13}\text{C}$  ( $\delta^{13}\text{C}$  average =  $0.42 \pm 1.96\text{‰}$ ) than lower layers, with an extreme range of values ( $5.35\text{‰}$  to  $-1.45\text{‰}$ ). If the extreme positive  $\delta^{13}\text{C}$  is excluded the average value is  $-0.13 \pm 0.97\text{‰}$  which is similar to those obtained on *A. antiquata* (average =  $-0.45 \pm 0.75\text{‰}$ ) in layers V to VII. There is no significant change in  $\delta^{18}\text{O}$  (average =  $-1.40 \pm 0.50\text{‰}$ ). Suspecting instrument drift, we re-sampled these shells and the isotope results were confirmed (Supplementary Table 3). This isotopic shift is short-lived and only found in Layer IV and only in the *Gafrarium* shells (Figure 3). The average  $\delta^{13}\text{C}$  value for *Anadara* from Layer IV is  $-1.22 \pm 0.87\text{‰}$ , which is slightly lower than shells from layers V to VII. Again, there is no significant change in  $\delta^{18}\text{O}$  ( $-0.88 \pm 0.54\text{‰}$ ). The two valves of *Quidnipagus* have similar isotope results to the upper layers ( $\delta^{13}\text{C} = -0.29 \pm 0.15\text{‰}$ ;  $\delta^{18}\text{O} = -1.07 \pm 0.14\text{‰}$ ).

Using the same “marine” and “estuarine”  $\delta^{13}\text{C}$  division as before, only one *Gafrarium* was clearly marine (Wk-45855,  $\delta^{13}\text{C} = 5.35\text{‰}$ ). One sample, (Wk-45684) was borderline ( $\delta^{13}\text{C} = 1.45\text{‰}$ ). The remaining eight had estuarine values. Wk-45855, Wk-45684, Wk-45861 ( $\delta^{13}\text{C} = -1.19\text{‰}$ ) and Wk-45862 ( $\delta^{13}\text{C} = -0.76\text{‰}$ ) were selected for dating and  $\Delta R$  calculation. None of the *Anadara* shells from Layer IV had marine  $\delta^{13}\text{C}$  signatures, but since *Anadara* from this layer had not previously been dated we selected two with  $\delta^{13}\text{C}$  extremes for comparison (Wk-45871,  $\delta^{13}\text{C} = 0.71\text{‰}$ ; Wk-45867,  $\delta^{13}\text{C} = -2.27\text{‰}$ ). Two *Tridacna* shells considered to represent marine conditions were also dated; Wk-45847 ( $\delta^{13}\text{C} = 2.13\text{‰}$ ) and Wk-45866 ( $\delta^{13}\text{C} = 2.81\text{‰}$ ). Only one charcoal value was available from Unit 4, Layer IV for comparison (SANU 2445 ± 31BP). The absence of a short-lived charcoal sample

for comparison is not ideal, but the  $^{14}\text{C}$  age is comparable to charcoal dates from similar contexts in neighbouring excavation units (Figure 8).

Three of the *Gafrarium* valves with estuarine  $\delta^{13}\text{C}$  values have elevated  $\Delta\text{R}$  (Wk-45861 =  $322\pm 76$ , Wk-45862 =  $195\pm 76$  and Wk-45864 =  $660\pm 76$   $^{14}\text{C}$  years). Similarly, the *Anadara* valves have  $\Delta\text{R}$  values of  $304\pm 76$  (Wk-45867) and  $533\pm 74$  (Wk-45871)  $^{14}\text{C}$  years. Two of these  $\Delta\text{R}$  offsets are much larger (*Anadara*; Wk-45871 and *Gafrarium*; Wk-45864) than those calculated for shells from layers V to VII. The cause of these two extreme values is difficult to evaluate. This variation, may reflect the unstable reservoir situation as sea-level dropped, mobilising old sediment and drainage of the Mariana Limestone aquifer, and would suggest some variability in the hardwater input at this time. However, while a hardwater offset can be evoked, evidence from the site suggests mixing with earlier (layers V to VII) activity is possible. Wk-45864 and Wk-45871 both come from 130-140cm depth; initial signs of disturbance were recorded in the field at the 140cm interface with material from Layer V. Upward displacement is consistent with material evidence elsewhere in the site. Excluding the two extremes the average estuarine  $\Delta\text{R}$  value ( $274\pm 44$ ;  $\chi^2_{2;0.05} = 1.64 < 5.99$ ; GSD=68.72) is comparable to that calculated for layers V to VII.

Two *Tridacna* have  $\Delta\text{R}$  offsets of  $-82\pm 76$  and  $-44\pm 75$   $^{14}\text{C}$  years. Combined with the “marine” *Gafrarium* sample (Wk-45855;  $-39\pm 75$   $^{14}\text{C}$  years) we calculate a value of  $-55\pm 44$   $^{14}\text{C}$  years ( $\chi^2_{2;0.05} = 0.19 < 5.99$ ; GSD=30.41) in-keeping with the marine correction value for layers V to VII. This suggests there has been no noticeable change in the open marine reservoir.

#### 4. The Chronology of Bapot-1

##### 4.1. Shell chronology with marine/estuarine $\Delta\text{R}$

Figure 6a highlights the problem of applying a uniform  $\Delta\text{R}$  correction (in this instance 0) to all shell dates from Bapot-1, Unit 4. In this example, the chronology of the earliest deposits is spread over c. 500 years with later deposits (Layer IV upwards) showing greater spread in ages. Without careful evaluation of the site and material culture it is possible to argue that these deposits are disturbed. However, if we apply the estuarine and marine  $\Delta\text{R}$  values to *Gafrarium* and *Anadara* shells based on  $\delta^{13}\text{C}$ , the chronology changes dramatically. Figure 6b shows the revised chronology of Unit 4 where Bayesian modelling has been undertaken. Here dates have been arranged in phases corresponding with the depositional layers (Bronk Ramsey 2009a). A sequential boundary separates layers IV and V corresponding to a gap in the  $^{14}\text{C}$  ages and the major ceramic change identified by Winter (2015). The internal consistency of the calibrated ages was tested using a General t-type Outlier Model that enables outliers to be either too young or too old, and down-weights their influence in the model (Bronk Ramsey 2009b). All dates were assigned a prior outlier probability of 0.05. Calibrated ages before the model parameters have been applied (“prior probability values”) are shown as unfilled outlines. Posterior probability values after the model has been applied are shown in black. All calibrated dates given in the text are reported at 95% probability unless otherwise stated.

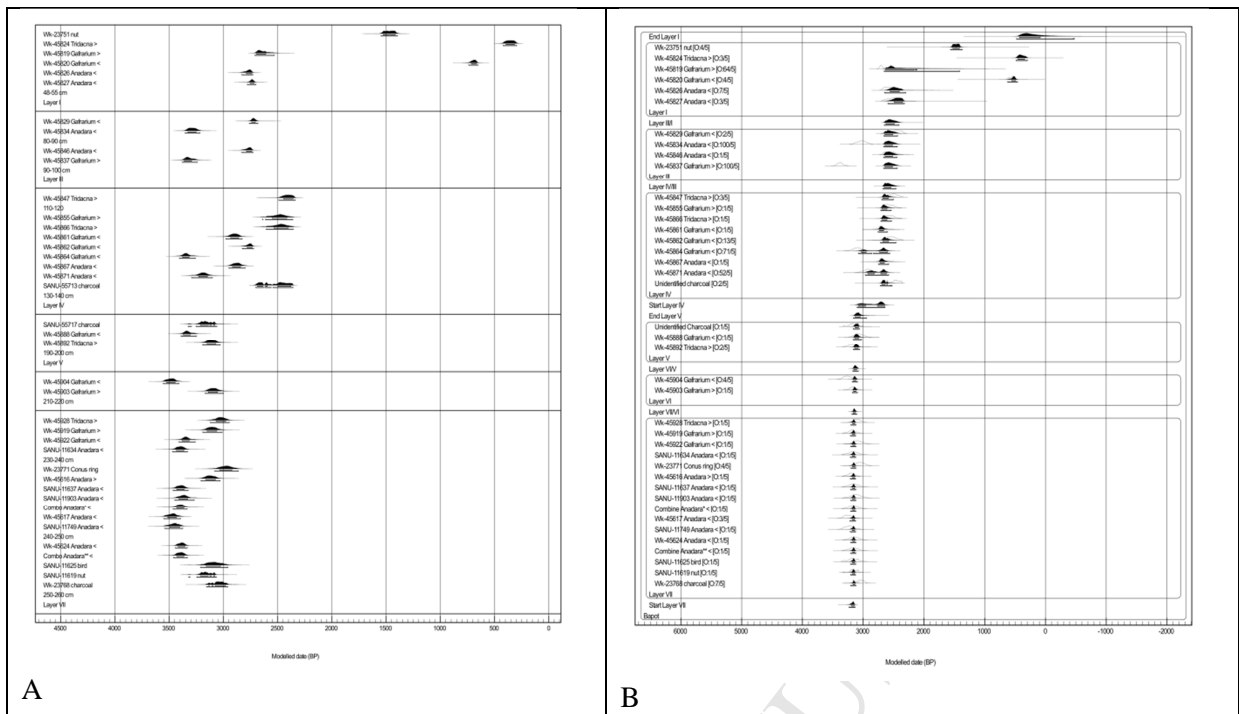
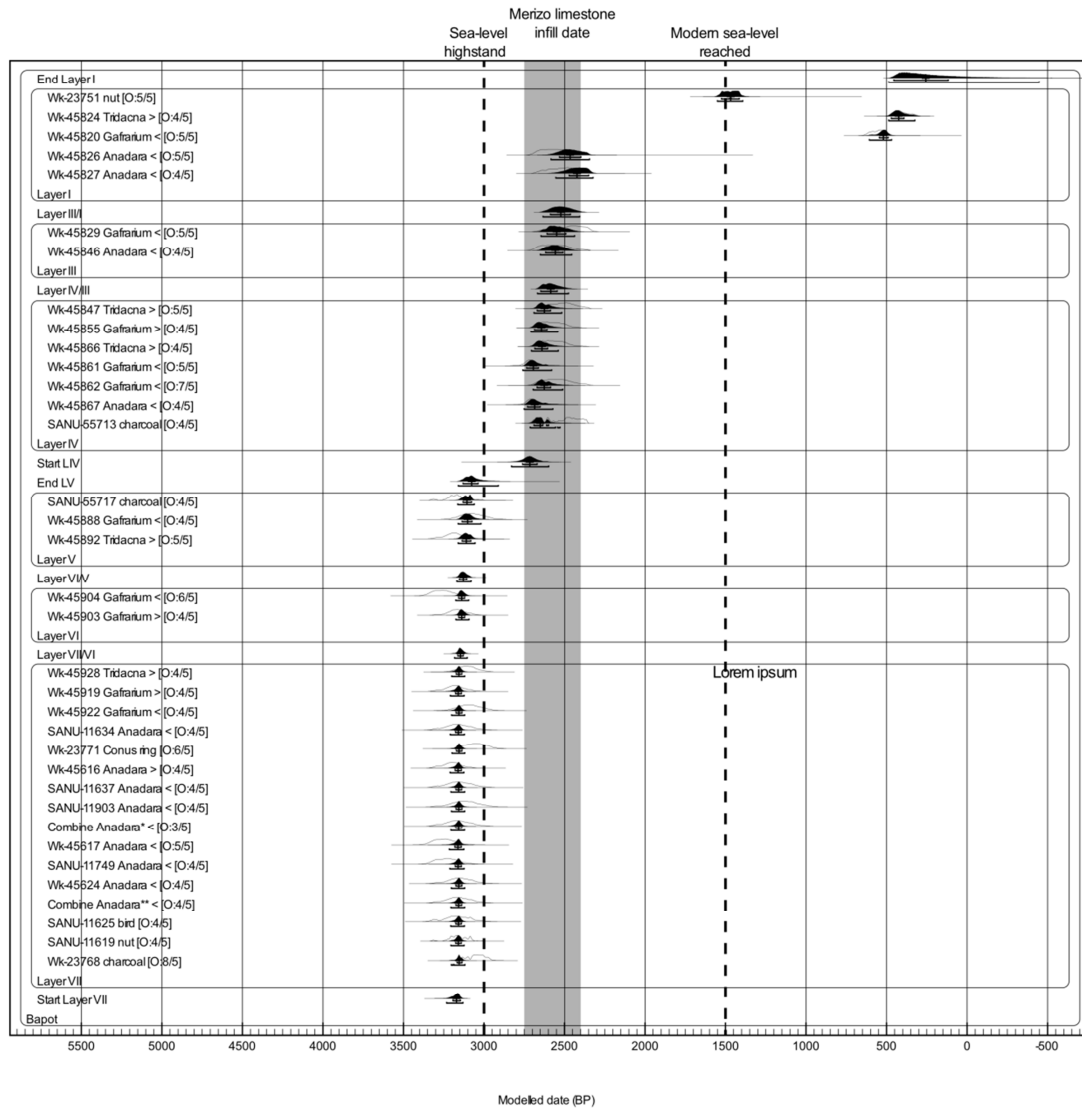


Figure 6. Charcoal and shell radiocarbon dates from Unit 4. A) Uncorrected for  $\Delta R$ . B) Bayesian sequence model showing shell calibrated ages using  $\delta^{13}\text{C}$  determined average “estuarine” (<) and “marine” (>)  $\Delta R$  values of  $197 \pm 43$   $^{14}\text{C}$  years and  $-55 \pm 27$   $^{14}\text{C}$  years. 68% and 95% error margins are indicated by bars under each age distribution. The notation [O:2/5] indicates a 2% posterior probability of being an outlier in the model. Combine Anadara\* = SANU-11901 + SANU-11748; Combine Anadara\*\* = SANU-11750 + SANU-11902.

In this model (Figure 6b), only charcoal date Wk-23768 from Layer VII is identified as an outlier (7%). This result is slightly younger than the associated shell and bird dates. Rather than reflecting stratigraphic displacement, this offset could be caused by the large number of shell ages influenced by hardwater. This result has minor impact on the model in these lower layers as indicated by high convergence values (>95%) for boundaries and individual dates generated by the OxCal MCMC algorithms. However, by Layer IV major issues with the chronology start to appear. Two major (>20%) outliers (Wk-45871; 52% and Wk-45864; 71%), and one minor outlier (Wk-45862; 13%) are identified (Supplementary Table 4). By Layer III two major outliers are excluded from the model 100% of the time (Wk-45837 and Wk-45834). These outliers all appear to be of similar age to the earliest shell dates and upward movement of objects is in-keeping with other material finds at the site. These outliers have a major impact on the convergence values for the boundary ages (End IV/Start III = 84.6%; End III/Start I = 84.9%) indicating many different incompatible solutions to the model at these points (Supplementary Table 4).

Removing the five major (>20%) outliers from the model (i.e., Wk-45819, Wk-45834, Wk-45837, Wk-45864 and Wk-45871) improves the convergence values (Supplementary Table 5). Overall, these results suggest two major periods of use (Figure 7 and Table 3); the first represented by layers VII, VI and V dated to 3240-2910 cal. BP, followed by a period of at least 140 years with no evidence of activity in this unit. The second period of activity is represented by layers IV and III, starting c. 2830 cal. BP and ending around 2400 cal. BP. Dates from Layer I display considerable variability with ages congruent with earlier layers as well as material dating to 1550-1390 cal. BP (Wk-23751) and 610-320 BP (Wk-45820, and Wk-45824).





**Figure 7:** Bayesian sequence model for Unit 4, Bapot-1 excluding major (>20%) outliers. Highstand and sea-level stabilisation dates based on Athens and Ward (2005). Merizo infill coral date = 2760-2400 cal. BP.

	Modelled calibrated age 68% probability	Modelled calibrated age 95% probability
Boundary Start Layer VII	3200-3140	<b>3240</b> -3130
Boundary Layer VII/VI	3170-3120	3190-3100
Boundary Layer VI/V	3160-3100	3170-3080
Boundary End Layer V	3130-3030	3160- <b>2910</b>
Interval	290 to 440-year gap	140 to 510-year gap
Boundary Start Layer IV	2760-2670	<b>2830</b> -2590
Boundary Layer IV/III	2650-2540	2670-2470
Boundary Layer III/I	2590-2460	2640- <b>2400</b>
Wk-23751 nut	1530-1410	1550-1390
Wk-45824 <i>Tridacna</i>	480-390	490-320

Wk-45820 <i>Gafrarium</i>	550-490	610-460
<b>Boundary End Layer I</b>	460-110	490-0

Table 3: Chronology for Unit 4, Block A excavations following exclusion of major (>20%) outliers.

#### 4.2 Charcoal chronology

If the chronological model for Unit 4 based largely on shell dates suggest the earliest occupation starts in the interval 3240-2910 cal. BP with renewed activity beginning around 2830 cal. BP, can this time gap be corroborated using other dating evidence from other excavation units at Bapot-1?

Figure 8 shows calibrated charcoal dates from the 2008 Block A excavations (includes unpublished charcoal dates (Clark unpublished data) as well as values reported in Petchey et al. [2017] and Clark et al. [2011]). These charcoal dates are grouped into four “phases” based on age and approximate stratigraphic relationship; the loose sandy soil, vertical movement of small samples, and possible inbuilt age in the charcoal, complicates the correlation between different excavation units. In this chronological model, three dates are identified as minor outliers with little impact on the model convergence values; Wk-23751 (6%), Wk-23752 (6%) and Wk-23760 (6%) (Supplementary Table 6). The earliest occupation starts 3290 cal. BP and ends by 2940 cal. BP, which corresponds to the modelled shell ages for layers VII to V. This is followed by a short hiatus of at least 320 years with renewed activity indicated after 2690 cal. BP. Subsequent activity starts 1890 cal. BP, and continues up until 510 cal. BP (modelled boundary ranges are given in Table 4).

This chronological pattern is similar to that modelled for Unit 4 which is based mainly on shell ages, except that the second phase of activity (represented by Layer IV) occurs after 2830 cal. BP (Figures 7 and 8). This additional c. 140-year difference between the shell and charcoal chronologies could be caused by an increase in the hardwater offset; two dates on *Tridacna* (Wk-45847 and Wk-45866) are the only non-estuarine shellfish dated from this layer and they give unmodeled  $\Delta R$  corrected ages of 2670-2370 cal. BP and 2700-2440 cal. BP. This lends support to a slightly later date for the second phase of activity and suggests that the recorded negative shift and increased variability in  $\delta^{13}C$  for *Gafrarium* shells in Layer IV reflects changes in the nearshore environment that have resulted in an under-correction for hardwater input at this time. This observation requires further testing.

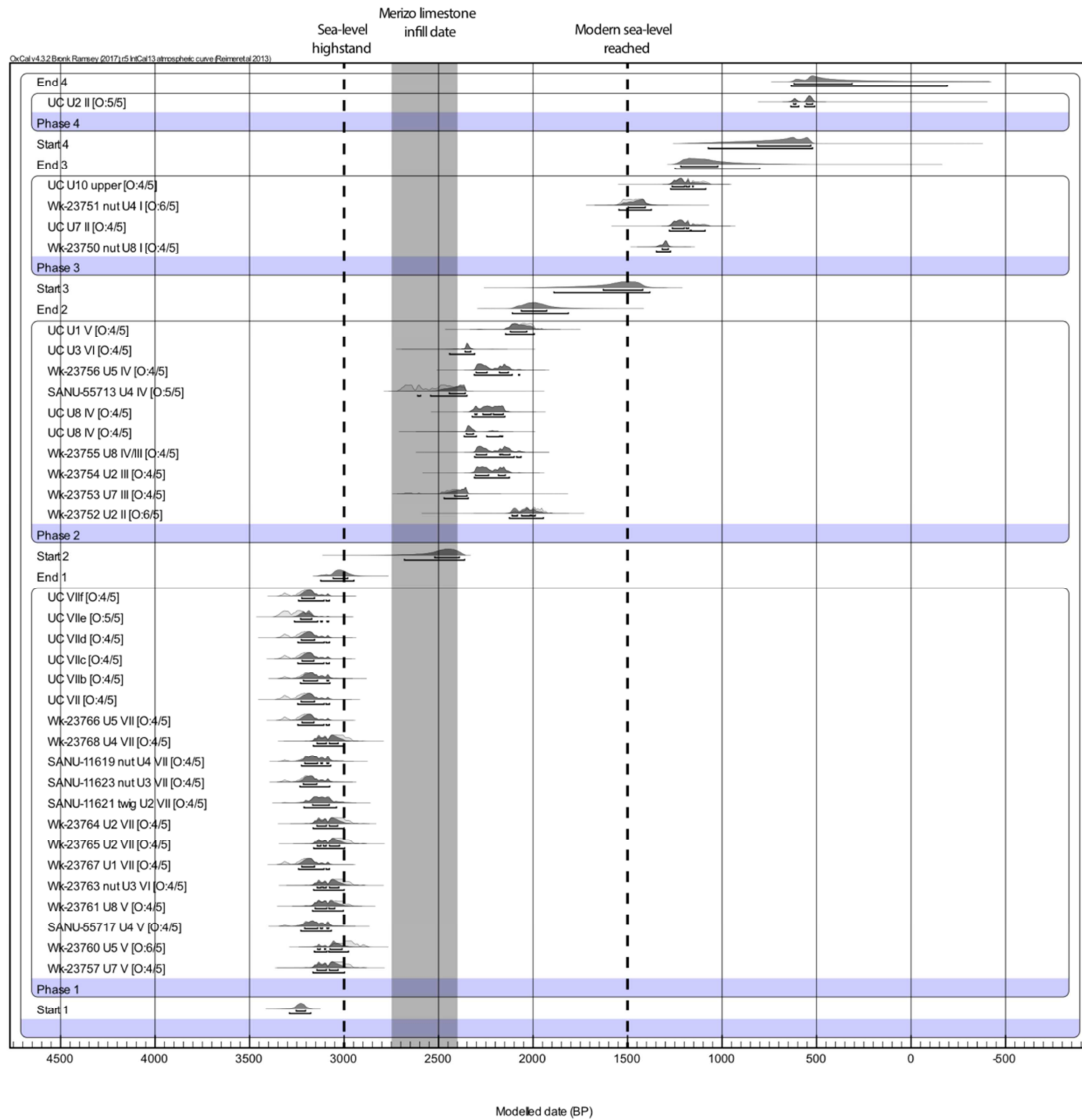


Figure 8: Charcoal  $^{14}\text{C}$  dates from Bapot-1, Block A excavations, divided into Phases 1-4. UC = unpublished radiocarbon dates; U = Unit; I-VI = layer designation.

	<b>Modelled calibrated age 68% probability</b>	<b>Modelled calibrated age 95% probability</b>
<b>Boundary Start 1</b>	3260-3200	<b>3290-3170</b>
<b>Boundary End 1</b>	3060-2980	<b>3130-2940</b>
<b>Interval</b>	470 to 650-year gap	320 to 720-year gap
<b>Boundary Start 2</b>	2520-2390	<b>2690-2360</b>
<b>Boundary End 2</b>	2070-1920	<b>2110-1810</b>
<b>Interval</b>	310 to 590-year gap	70 to 650-year gap
<b>Boundary Start 3</b>	1630-1410	<b>1890-1380</b>
<b>Boundary End 3</b>	1220-1020	<b>1250-800</b>
<b>Interval</b>	160 to 560-year gap	0 to 610-year gap
<b>R_Date UC U2 II</b>	630-520	<b>640-510</b>

Table 4: Modelled charcoal chronology for all charcoal dates obtained from the Bapot-1 Block A excavations.

## 5. Discussion

This research has a number of significant findings of interest to those studying the age of first colonisation of Remote Oceania, and to researchers worldwide who use shells for chronological control:

1. Our results indicate that  $^{13}\text{C}$  is a useful tool to differentiate between marine and estuarine influence on tropical filter-feeding bivalves. At Bapot-1 these  $\delta^{13}\text{C}$  differences are likely to be caused by uptake of DIC derived from decayed plant matter within the mangrove environment that *Anadara* and *Gafrarium* favour. Conversely, reef bivalves such as *Tridacna* sp. primarily incorporate DIC from ocean water. Our research suggests a minimum value of 1.7‰ to separate estuarine and marine influence in the Bapot-1 shells. One *Gafrarium pectinatum* sample from Layer III (Wk-45837;  $\delta^{13}\text{C} = 1.90\text{‰}$ ,  $3442 \pm 15$  BP) does not conform to the proposed  $\delta^{13}\text{C}/^{14}\text{C}$  division. If a marine  $\Delta\text{R}$  correction is applied the resultant calibrated age would make it the oldest shell recovered (3480-3290 cal. BP). We conclude that this sample is most likely displaced from layers V to VII or from a natural source.
2.  $\delta^{18}\text{O}$  is a useful indicator of major freshwater input, but interpretation is complicated by temperature fluctuations in these near-shore environments.
3. An apparent estuarine  $\delta^{13}\text{C}$  value for shellfish, even in limestone environments, does not necessarily guarantee a hardwater effect – this has to be based on the weight of geological, archaeological and hydrological evidence as well as the ecology of the shells dated. Moreover, in regions where there is no limestone, 5% terrestrial (modern) carbon (resulting in an offset of c. -20 years) would be masked by the date precision.
4. Based on shell isotopes and geological and hydrological observations, it is probable that “hardwater” has influenced the Bapot-1 estuarine shellfish. This conclusion remains unchanged from that given in Petchey et al. (2017). Because mangroves hold any freshwater discharge from the aquifer close to the coastline it is likely that water conditions will be highly specific to the immediate environment (Miklavic 2011:40). This, combined with the mix of limestone and volcanic geologies across the Mariana Islands and variable drainage (Mink & Vacher 1997; Stafford et al. 2005), negates a uniform  $\Delta\text{R}$  value across the region for any shellfish in the estuarine zone.
5. Open marine filter-feeding species such as *Tridacna*, which do not tolerate freshwater, have a  $\Delta\text{R}$  ( $-55 \pm 27$   $^{14}\text{C}$  years) that differs little from the Pacific average of  $-84 \pm 69$   $^{14}\text{C}$  years for the period between 2500 and 3000 cal. BP (Komugabe-Dixson et al. 2016). Our research suggests  $\Delta\text{R}$  will be more uniform for bivalves that prefer marine habitats, but this has yet to be tested across multiple islands.
6. We calculate an average “estuarine”  $\Delta\text{R}$  offset of  $197 \pm 43$   $^{14}\text{C}$  years at c. 3200 cal. BP. This 250-year difference between estuarine and open ocean (marine) shells is equivalent to c. 3% input of ancient DIC into the Lao Lao Bay. By c. 2690 years ago there is a change in the estuarine reservoir as indicated by shifting  $\delta^{13}\text{C}$  of *Gafrarium* shells and an increased offset between charcoal and *Anadara* and *Gafrarium* dates. This ties in with a  $^{14}\text{C}$  date on the Merizo limestone infill interpreted by Athens and Ward (1999) to indicate that reef building had declined and coastal infilling had begun following a rapid drop in sea-level.
7. We have identified little change over time in the *Anadara antiquata*  $\delta^{13}\text{C}$  values. *Anadara* sp. do not tolerate change (Davenport and Wong 1986) and if they quickly die once the environment changes as hypothesised, it makes sense that they will disappear before isotopic signals of change can be detected. By Layer IV the numbers of *Anadara* are dropping and by Layer III they are largely extirpated from this location, though occasional harvesting is still possible as indicated by ANU-4771 ( $1040 \pm 110$  BP;  $\delta^{13}\text{C} = 0.9\text{‰}$ ) reported by Bonhomme and Craib (1987).

8. *Gafrarium* sp. are more tolerant of changing environmental conditions and have a higher tolerance to low salinity waters (McMahon 2003) so should give us a greater potential for tracking change in the nearshore environment. *Gafrarium pectinatum*  $\delta^{13}\text{C}$  values from Layer IV are on average more negative and varied than lower deposits, indicating a significant change to their habitat after 2830 years ago; a time when they begin to dominate over *Anadara*. Similar shifts towards more negative  $\delta^{13}\text{C}$  values have been attributed to change in vegetation type (Surge et al. 2003), the decay of phytoplankton (Hong et al. 1996), or changes to local water source (Swart et al. 1996, Surge et al. 2003). Theoretically, the loss of mangrove habitats caused by declining sea-level should have resulted in a movement to more positive  $\delta^{13}\text{C}$  values as mangroves ( $\delta^{13}\text{C} = -27\text{‰}$ ) were replaced by seagrass ( $\delta^{13}\text{C} = -16.3$  to  $-7.3\text{‰}$ ) (values taken from Surge et al. 2003). This would result in a gradual change to isotopic conditions in the nearshore environment. Addition of quantities of freshwater could result in eutrophic conditions due to the decomposition of plant material, but seems less plausible in the wide-open Lao Lao Bay. If there was a significant change in the amount of rainfall or drainage of the limestone aquifer, a concomitant shift in  $\delta^{18}\text{O}$  would be expected, which is not the case for the shells from Bapot-1. However, discrepancy between estuarine shell and charcoal dates c. 2500 years ago (Layer IV/Phase 2) supports the hypothesis of changing nearshore conditions.
9. There is disturbance at Bapot-1 but this is restricted to the upper spits of Layer V and above and does not appear in the artefact distribution or shellfish ages until Layer IV.
10. A comparison of shell and charcoal dates suggests earliest use of the site started after 3290 years ago, similar to the 3230–3085 cal. BP (95% probability) earliest settlement age calculated by Rieth and Athen's (2017) for the Mariana Islands as a whole. After a short hiatus of at least 140 years this was followed with renewed activity at Bapot-1 during a time when the offshore reefs were declining and coastal infilling was underway as sea-level fell rapidly (Figures 7 and 8).
11. The use of  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$  evaluation before  $^{14}\text{C}$  measurement is more cost-effective than the usual hit and miss process of dating shellfish based on habitat and species assumptions, especially in problematic environments affected by hardwater. Stable isotopic data also adds to our understanding of processes impacting on the people living at the site and is a valuable addition to the archaeological interpretation.

## 6. Conclusion

Our research has reaffirmed that shells with known estuarine ecologies, such as *Anadara antiquata* and *Gafrarium pectinatum*, could be affected by sources of terrestrial carbon which could adversely affect Pacific archaeological chronologies. The suitability of these shellfish for  $^{14}\text{C}$  dating can, however, be assessed using  $\delta^{18}\text{O}$  and  $\delta^{13}\text{C}$ . In areas where limestone is present, selecting shells that reflect oceanic isotopic conditions provides the best means to obtain reliable ages.

Isotopic and species habitat and dietary information combined with hydrological, geological and oceanographic information support the conclusions of Petchey et al. (2017) that hardwater affected both *Anadara* and *Gafrarium* shells from the early contact site of Bapot-1 on the island of Saipan. This late chronology has multiple and far-reaching implications for our theories about population origins, movement and health, technological adaptation, domestication, and environmental impact throughout Oceania. With appropriate reservoir corrections for marine and estuarine  $^{14}\text{C}$  reservoirs, both charcoal and shell chronologies for Bapot-1 are brought into congruence and indicate that first settlement occurred after 3290 years ago, followed by a short hiatus and renewed activity after 2690 years ago.

Evaluation of the  $\delta^{13}\text{C}$  of different shellfish taxa over time has also enabled us to recognise a significant change to the nearshore environment starting c. 2690 years ago, most likely associated the loss of mangrove habitats and draining of freshwater from the island limestone aquifer, as sea-level fell during the mid-Holocene. While most shellfish found these conditions intolerable, *Gafrarium pectinatum* were able to survive and become the dominant shellfish gathered, providing a means by which we can now track changes over time in the nearshore environment.

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## HIGHLIGHTS

1. The suitability of estuarine shells for  $^{14}\text{C}$  dating is assessed using  $^{18}\text{O}$  and  $^{13}\text{C}$ .
2. We calculate a marine  $\Delta R$  of  $-55 \pm 26$   $^{14}\text{C}$  years for the region c. 3300 years ago.
3. Hardwater had caused c. 250 years error in estuarine shellfish.
4. The settlement of the Mariana Islands had taken place by 3290 cal. BP.
5. By 2690 years ago estuarine conditions change due to lowering sea-level.