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Radiocarbon dating in Petra: limitations and potential in semi-arid environments

J.F. Saliège, Antoine Zazzo, Christine Hatté, Caroline Gauthier

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Men on the Rocks

The Formation of Nabataean Petra

M. Mouton & S. G. Schmid (editors)

Men on the Rocks

The Formation of Nabataean Petra



Proceedings of a conference held in Berlin
2–4 December 2011

M. Mouton & S. G. Schmid (editors)



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Radiocarbon dating in Petra: limitations and potential in semi-arid environments

Jean-François Saliège †, Antoine Zazzo,
Christine Hatté & Caroline Gauthier

As part of the chronological study of the urban site of Petra, we have compared ^{14}C dating derived chronology with the pottery and coin assemblages from the site. In order to minimise the risk inherent in using only one type of ^{14}C material, we chose to date all the material possible for each phase. It is the convergence of the results that ensures coherence in the dating. The main difficulty comes from uncertainties in the calibration of ^{14}C dates. In spite of this drawback, the combination of archaeological and geochemical data allowed to date the Hellenistic levels (Phase I) of the urban site of Petra back to the 4th century BC. The construction of dwellings in phase II must be dated to the 3rd century BC, their occupation continuing into the 2nd and 1st centuries BC.

PRESENTATION

The French archaeological missions in Petra carried between 2004 and 2009 allowed to propose a chronology for the Hellenistic levels, based on the pottery and coin assemblages (Renel *et al.* 2012). During the excavations, material was recovered that was datable using the radiocarbon method (^{14}C). One part of the French-German ANR funded “Early Petra” project consisted of comparing the ^{14}C dates and the archaeological data. Radiocarbon dating is the most precise physico-chemical dating method and can provide reliable dating information in this context, on condition that the limitations of each type of source material are identified. Furthermore, this method is all too often perceived by excavators as being independent of location and sampling conditions. However, the climatic factor is essential in the choice and conservation of samples. From

this point of view, there is a difference between temperate regions, and arid and semi-arid areas.

In the last thirty years, there has been much progress in ^{14}C dating thanks to accelerator mass spectrometry (AMS). The method is no longer limited by issues of quantity (0.5 to 1 mg of carbon is sufficient for a routine AMS measurement), which increases its scope. The use of materials from short-lived sources is being developed. It is now possible to consider using remains of seeds, fruit, basketry, cloth and pollen from archaeological strata (e.g. Smith 1997, Caran *et al.* 2001, Piperno & Stothert 2003, Bourke *et al.* 2007). In exceptional conditions, AMS can also date pure lime, such as surface plastering or mortar (e.g. Folk & Valastro 1976, Zouridakis *et al.* 1987, Heinemeier *et al.* 2010) as well as organic inclusions in mortar (mainly charcoal and straw) (e.g. Berger *et al.* 1992, Rech *et al.* 2004, Al-Bashaireh & Hodgins 2011). Organic matter included in and on some pottery can also be dated. However, the use of these materials is limited. Charcoal and bones (collagen and bioapatite in some circumstances) remain the most accurate sources for dating archaeological sites. The ^{14}C material contained in archaeological layers that is found during excavation is simply one object amongst others and is subject to the same stratigraphic uncertainties. The precision of AMS measurements is generally in the order of ± 20 to ± 40 years for the Holocene (Bronk Ramsey *et al.* 2004). Dendrochronological calibration means that one can move from a ^{14}C chronology into historical periods (Bronk Ramsey 1995, Reimer *et al.* 2004), and in certain circumstances, Bayesian analysis can refine these clusters of dates (Bronk Ramsey *et al.* 2010). The main challenge now is

both to identify the limitations of the ^{14}C material itself and its relevance within the archaeological and environmental context.

ADVANTAGES AND LIMITATIONS OF ^{14}C MATERIALS IN SEMI-ARID CONDITIONS

Charcoal

Tree rings accumulate from the center to the periphery of the tree trunk, so that the ^{14}C activity of the first ring gives the time elapsed since the beginning of the tree life, whereas ^{14}C dating of the last ring will indicate the time elapsed between the death of the tree and its use. In arid areas some trees such as olive trees, juniper etc. are very long-lived. This can lead to a large difference in age between the centre and the external part of the trunk. Moreover, in these regions, wood is rare and thus is often re-used. Excavators look for charcoal and wood mainly as evidence of hearths, tools or wooden structures. It should be emphasised that the classic techniques of ^{14}C measurements (proportional counters and liquid scintillation) need about 1 or 2 grams of carbon. Such quantities are usually found in hearths and the average age measured is often representative enough of the age of the archaeological level. ^{14}C measurement by AMS reduces the size of sample needed by a factor of one thousand. However, with this considerable advantage comes the increased risk of sampling charcoal from the centre of a tree trunk, which would result in an overestimate of the age of the hearth. This overestimation could simply be the result of the collection of dead wood, but also, from the start of the construction of timber framed structures, it seems that in the Middle East, such building wood was frequently reused, thus introducing potential errors in the dating (Saliège & Pessin 2005). Charcoal can therefore only give a *terminus post quem* for the layer being dated. Thus, it is tempting to look for the carbonised remains of short-lived materials such as twigs and seeds. But the remains of twigs are often confused with the deep roots of surface vegetation and isolated seeds could be deposited by burrowing animals.

Unburned bones

Radiocarbon dating of human or animal bones is of major interest to archaeologists. In arid environments, the climatic conditions do not usually favour the preservation of collagen, which is rapidly hydrolysed. Thus, the mineral fraction of

bones and teeth, the hydroxyapatite or bioapatite carbonate, is often the only option for dating sites in these environments, as long as diagenetic changes have not occurred. The bioapatites (particularly bones and dentine) are porous tissues which can behave as an open biochemical system. They are sensitive to the hydrological environment (Hedges & Millard 1995, Wright & Schwarcz 1996, Zazzo *et al.* 2004). The preservation of bioapatites will thus depend to a large extent on the climatic conditions during fossilization. The validity of ^{14}C dates from human bioapatite was tested successfully during the excavations of the large necropolises of Air and Adrar Bous in Niger, which was the first significant geochemical study of bone from an arid environment (Saliège *et al.* 1995). The review paper by Zazzo & Saliège (2011) shows that overall, in the Holocene at least, bioapatite from arid and semi-arid areas is resistant to diagenetic changes.

Heated bones

To the mineral fraction of non-heated bones, we can add the remaining organic matter from burnt bones. Heating bones to a temperature up to $400\text{ }^{\circ}\text{C}$ partly fixes the collagen by decomposing it. This degraded organic matter contains carbon and can be dated. Experiment shows that this organic carbon, extracted during pre-treatment, is sensitive to contamination (Higham *et al.* 2011). About ten years ago, carbonate from calcined bone was suggested as a source of carbon that could be reliably dated by ^{14}C (Lanting *et al.* 2001). Above $600\text{ }^{\circ}\text{C}$, the mineral fraction of the bone recrystallises and becomes less soluble, thus providing better protection against the alteration of the residual carbonates in the bone. This property has since been verified on many archaeological sites, and it seems that calcined bone dating is reliable, at least for the Holocene (Zazzo & Saliège 2011). More recently, laboratory and field experiments have shown that during heating, bone behaves as an open biochemical system with regard to the carbon dioxide in its immediate environment (atmospheric CO_2 and / or CO_2 produced during the combustion of wood). During calcination, the bone incorporates carbon from these two sources into its mineral structure, in the form of carbonate, and the original carbon in the bone then represents only a minor fraction (20 to 30%) (Zazzo *et al.* 2012). The carbon that is dated is thus mainly a mix of these two CO_2 sources. In arid environments, where wood is quite rare, one

can predict an apparent increase of the age of the bone in a case where old wood might have been used to burn it, although this hypothesis has not yet been tested.

Pottery

Numerous laboratories have worked on refining physiochemical methods for dating pottery which is a primary material for dating in archaeology. The best-known method is thermoluminescence (Aitken 1985). Since the inception of ^{14}C dating, there have also been sporadic attempts to date organic material included in and on pottery (e.g. Ralph 1959, Stukenrath 1963, Atley 1980, Johnson *et al.* 1986, Gabasio *et al.* 1986). The main problem comes from the varied origin of the sources of organic carbon included in and on sherds. By reconstructing the life of a pottery vessel, one can define the different elements (a, b, c, d, e, f, g, h) as follows. To begin with, the potter chooses a raw material of clay sediment because of its plastic qualities. This material contains an organic fraction which has no direct relation with the age of the pottery: this is the first source of carbon (a). In order to avoid shrinkage cracks in the clay during firing, the potter adds a mineral temper or a temper composed of organic and / or plant remains (b) to the clay paste. Then, during the firing, the fuel can deposit smoke carbon on surfaces. The pyrolysed carbon from this source can diffuse through the clay paste, and in poorly fired pottery remains on the surface (c). After the chemical transformations due to heat (in particular, a heavy reduction of the organic carbon content) the craftsman might coat the vessel with an organic slip (d). Then, during domestic use, organic food residues might be deposited on the inside surface during cooking (e) or through storage of food products (such as wine or oil) (f). The outside surface of cooking pots is sometimes covered with carbonaceous deposits from the hearth (g). Finally, during deposition, sherds can be contaminated over time, by soil organic acids and by the rootlets of the surface vegetation (h). All these sources of carbon, with the exception of the carbon in the clay (a) and the contaminants (h) are, with a few exceptions, contemporary with the fabrication and use of the pottery vessel. Stating these different elements suggests a relatively simple methodology for obtaining reliable dates with the help of AMS. Reality is more complex, however. Dating plant temper is only applicable to poorly fired vessels,

which, in practice, means to Neolithic and proto-historic vessels. In Europe, the nature of organic tempers (b) is varied: crushed bone, moss, plants etc. (e.g. Constantin & Courtois 1985, Constantin & Kuijper 2002), and the residual organic carbon content after firing is relatively low (Delqué-Kolic 1995). Organic food deposits (e) found on some sherds have been dated by isolating the lipids (Hedges *et al.* 1992, Stott *et al.* 2003, Berstan *et al.* 2008). Firing smoke carbon (c) has been studied, but dating it comes up against the problem of organic pollution linked both to fine soil particles which stick to the surface of the sherds and the organic acids in the environment. In arid and semi-arid areas, the presence of grasses with so-called “C4” photosynthesis allows, through the use of their stable isotope content, a better understanding of the different organic sources included in and on the pottery (Saliège & Person 1991). Pottery vessels with high plant content have been dated in the Sahara (Serenio *et al.* 2009) and those with a temper of organic remains in the African Sahel (Hatté *et al.* 2010). In spite of the methodological problems, dating pottery has a good potential because it supplements the typology.

MATERIALS AVAILABLE IN PETRA AND ANALYTICAL METHODOLOGY

In Petra, we have dated charcoal, teeth and bones, and the organic matter included on and in pottery sherds. All these samples were subjected to pre-treatment in order to remove any contamination:

- Wood charcoal is able to absorb a lot of organic acids from the soil, which requires a classic pre-treatment of acid–alkaline–acid (1M HCl, 0.1M NaOH, 1M HCl) in order to eliminate contamination from carbonate and organic acids from the environment. In semi-arid areas, in the absence of soil the production of organic acids is low, which limits the risk of residual contamination after pre-treatment. The CO_2 was extracted by combustion at 800°C in the presence of O_2 .
- Tooth enamel was cleaned using a dentist’s drill to remove the dentine, and crushed to a grain size finer than $160\ \mu\text{m}$. Then the secondary carbonates were eliminated using acid in a vacuum (1M CH_3COOH). The CO_2 was extracted using phosphoric acid in vacuum, at 70°C .
- Degraded collagen underwent an AAA pre-treatment similar to that used on the charcoal. The collagen was extracted according to the method established by Longin (1971) and modified by

Bocherens (1992). The CO₂ was extracted by combustion at 800°C in the presence of O₂.

• For each sherd, we sampled a slice in section of a few millimetres thick. The samples then underwent a pre-treatment with HCl 1M at ambient temperature. The CO₂ was extracted by combustion at 600°C in the presence of O₂.

The CO₂ produced by the different samples was graphitized in order to measure the ¹⁴C / ¹²C ratio, using AMS at NSF-Arizona-AMS-Laboratory of Tucson, University of Arizona (Jull *et al.* 2004).

RESULTS

All the ¹⁴C materials have been reviewed above. In Petra, in order to reduce the risk inherent in using a single material type, we chose to date all the ¹⁴C materials possible for each phase. The convergence of results ensures coherent dating, or otherwise highlights any problems. With few exceptions, the accuracy of our measurements lies between ± 20 and ± 40 BP, but the calibration for these periods introduces a significant degree of uncertainty (Reimer *et al.* 2004). One must be particularly vigilant and evaluate each type of material to know whether, by its nature or its stratigraphic position, it might introduce an additional error. But before dating the Hellenistic levels, which was the aim of our work, we wanted to test our method on some well-dated soundings (table I).

We chose four teeth (bioapatite), one bone (collagen) and three pieces of charcoal. The two Nabataean-period teeth were dated using the enamel. The caprine tooth from level C4178 produced a date of 1904±31 ¹⁴C yr BP (AD 68–138, 1σ). The camel tooth from the same level was said by the excavators to belong to the late Nabataean. It returned a date of 1813±30 ¹⁴C yr BP (AD 138–239, 1σ). These two dates are acceptable even though they may appear slightly too recent due to the insufficient elimination of diagenetic carbonate in the enamel. The third, caprine, tooth is attributed to the late Roman period and gave a date of 2419±33 ¹⁴C yr BP. It is clear that only an error of stratigraphy can explain this aberrant date. The fourth, camel, tooth (C4116) assigned to the Roman period, produced an expected date of 1782±20 ¹⁴C yr BP (AD 219–321, 1σ).

A dating on collagen was attempted on a skeleton from an Abbasid grave. The date of 1180±120 ¹⁴C yr BP (711–974 AD, 1σ) is acceptable despite the high uncertainty linked to the very small amount of preserved collagen.

Finally, we chose charcoal from a burnt layer located in the Qasr al-Bint temple at the level of the western periobolos. In level C3110, 8 cm above the level of the paving of the periobolos, we obtained two dates of 1873±59 ¹⁴C yr BP (AD 77–214, 1σ) and 1868±34 ¹⁴C yr BP (AD 84–210, 1σ), and for the upper level 35–40 cm above the pavement, a date of 1877±75 ¹⁴C yr BP (AD 61–231, 1σ). The three dates, obtained from charcoal from the burnt layer in the soundings, are statistically identical. In spite of the good homogeneity of these dates, the age of the wood introduces a bias that is difficult to estimate and a more recent date for the fire cannot be excluded.

¹⁴C dating of the Hellenistic levels

Phase I

Eight dates were obtained, of which five were from charcoal coming from levels C9085-2, US 4251, US 4247 and US 4246, one from a root (C9085-1), one from degraded collagen from a bone of unknown origin (C9044), and one from ovicaprine enamel (US 4030) (table II, fig. 1). In these periods, the shift from the ¹⁴C chronology to calibrated dates extends the date range to a greater or lesser degree. Five dates can be counted in phase I as it is defined by the archaeologists, but two of the charcoal samples and the root fall outside this framework (C9085-1, 4246 and 4247a). The root, C9085-1, produced a date after 1950 AD. It could easily be thought that this little black root was part of the stratigraphy, but such was not the case. This example confirms the dangers of using very small samples. The charcoal (US 4246 and US 4247a) indicates calibrated dates that also fall outside the chronological frame. The charcoal from US 4246 (759–550 BC, 1σ), was found in isolation under a stone perhaps belonging to an ancient hearth. The charcoal from US 4247a and b (724–413 and 405–381 BC, 1σ) comes from a continuous ashy and charcoal layer (fig. 2). It is possible that the charcoal from US 4247a came from old wood. We note, however, that the highest probability (0.81, 1σ) for the charcoal from the ash layer is between 541 and 413 cal BC, not too far from the cluster of dates around the beginning of the 4th century BC. The date from the ovicaprine enamel (2153±39 ¹⁴C yr BP that returns two major calibrated 1σ intervals 210-157 BC and 352-296 BC) is the most recent from phase I, which might be linked to the poor removal of secondary carbonates present in enamel bioapatite. This date

Table 1 : Radiocarbon dates obtained in the Nabataean and Roman levels

prep	AMS	description	fraction	phase	US	$\delta^{13}C\%$	^{14}C age BP	68.2%		95.4%			
								from	to	from	to	proba	
Muse 14	AA90694	ovi-caprinae	enamel	Nabataean	C 4178	-7.3	1904 ± 31	68 AD	128AD	25 AD	43 AD	1.00	0.04
Muse 15	AA90695	camel	enamel	late Nabataean	C 4178	-5.9	1813 ± 30	138 AD	160 AD	126 AD	259 AD	0.24	0.95
GifA- 10 272	AA88862	charcoal	-	Roman	C 3110, 8 cm	-22.8	1873 ± 59	77 AD	214 AD	3 AD	258 AD	1.00	0.98
GifA- 10 273	AA88863	charcoal	-	Roman	C 3110, 8 cm	-23.2	1868 ± 34	84 AD	143 AD	72 AD	232 AD	0.62	1.00
GifA- 10 274	AA88864	charcoal	-	Roman	C 3108, 35-40 cm	-22.8	1877 ± 75	61 AD	231 AD	41 BC	263 AD	1.00	0.94
Muse 16	AA90696	camel	enamel	Roman	C 4116	-8.4	1782 ± 20	219 AD	258 AD	139 AD	196 AD	0.69	0.14
Muse 17	AA90697	ovi-caprinae	enamel	late Roman	C 2006 / 2000	-4.9	2419 ± 33	697 BC	696 BC	278 AD	329 AD	0.31	0.54
P996	AA90699	human tooth	collagen	Abbassid	grave 12	-22.9	1180 ± 120	538 BC	407 BC	666 BC	643 BC	0.99	0.04
								766 AD	974 AD	1089 AD	1122 AD	0.87	0.02
										1139 AD	1149 AD		0.01

Radiocarbon dates were calibrated using Calib Rev 5.0.1 (Reimer *et al.* 2004)

Table II : Radiocarbon dates obtained in the Hellenistic levels

prep	AMS	description	fraction	phase	US	context	%C	$\delta^{13}C\%$	^{14}C age BP	68.2%		95.4%		proba
										from	to	from	to	
Muse 11	AA90688	ovi-caprinae	enamel	II	US9040	rich organic layer	-	-9.3	2162 ± 21	348 BC	317 BC	355 BC	287 BC	0.48
Muse 12	AA90689	ovi-caprinae	enamel	II	US 10021	filling of a pit	-	-8.2	2194 ± 32	207 BC	174 BC	233 BC	157 BC	0.49
P998	AA90690	ovi-caprinae	enamel	II	US 10021	filling of a pit	-	-22.4	2247 ± 21	234 BC	200 BC	369 BC	177 BC	0.02
P1041	AA92554	burnt bone	dentin collagen	I-II	C3518	wall foundation	-	-17.3	2227 ± 40	356 BC	285 BC	389 BC	350 BC	0.35
P988	AA90693	burnt bone	decomposed collagen	I-II	C 9080	wall foundation	-	-15.4	2268 ± 21	285 BC	254 BC	304 BC	209 BC	0.65
Muse 13	AA90691	ovi-caprinae	enamel	I-II	C 9080	wall foundation	-	-10.3	2332 ± 32	249 BC	234 BC	292 BC	230 BC	0.41
P1039	AA92552	ovi-caprine	enamel	I	US 4030	on the virgin soil	-	-9.1	2153 ± 39	373 BC	350 BC	387 BC	201 BC	1.00
GifA-10269	AA88859	charcoal	-	I	C9085	terrace wall	-	-21.2	2278 ± 39	304 BC	209 BC	428 BC	360 BC	0.85
										275 BC	259 BC	217 BC	215 BC	0.01
										408 BC	383 BC	510 BC	434 BC	0.13
												273 BC	260 BC	0.02
										352 BC	296 BC	359 BC	275 BC	0.34
										228 BC	221 BC	260 BC	89 BC	0.64
										210 BC	157 BC	75 BC	57 BC	0.03
										135 BC	115 BC			
										397 BC	357 BC	402 BC	349 BC	0.47
										284 BC	256 BC	315 BC	208 BC	0.53
										247 BC	234 BC			

GifA-10271	AA88861	charcoal	-	I	US 4251	pit with virgin soil	-	-19.4	2292 ± 42	401 BC 282 BC 244 BC	357 BC 257 BC 235 BC	0.69 0.24 0.07	409 BC 319 BC	347 BC 207 BC	0.54 0.46
P1040	AA92553	burnt bone	decomposed collagen	I	US 9044	on the virgin soil	-	-20.3	2318 ± 40	410 BC 273 BC	360 BC 261 BC	0.92 0.08	510 BC 428 BC 295 BC 220 BC	434 BC 352 BC 228 BC 211 BC	0.10 0.71 0.18 0.01
GifA-10267	AA88857	charcoal	-	I	US 4247b	ashy layer	-	-21	2322 ± 32	405 BC	381 BC	1.00	486 BC 451 BC 418 BC 287 BC	460 BC 440 BC 356 BC 233 BC	0.02 0.01 0.88 0.08
GifA-10266	AA88856	charcoal	-	I	US 4247a	ashy layer	-	-21.4	2435 ± 32	724 BC 656 BC 541 BC	694 BC 656 BC 413 BC	0.18 0.01 0.81	751 BC 667 BC 621 BC 595 BC	686 BC 638 BC 614 BC 405 BC	0.22 0.07 0.01 0.70
GifA-10265	AA88855	charcoal	-	I	US 4246	ashy layer	-	-22.8	2495 ± 22	759 BC 689 BC 670 BC 647 BC	742 BC 683 BC 663 BC 550 BC	0.13 0.04 0.05 0.77	769 BC 696 BC	698 BC 538 BC	0.23 0.77
GifA-10276	AA88865	pottery	surface	I	US 11017	-	2.8	-11.3	1595 ± 44						
GifA-11182	AA94595	pottery	black matrix		US 11017	-	1.1	-20.2	1750 ± 60						
GifA-10277	AA88866	pottery	black matrix		US 11017	-	-	-21	2288 ± 47	401 BC 285 BC 250BC	356 BC 253 BC 234 BC	0.58 0.29 0.12	410 BC 330 BC	337 BC 203 BC	0.48 0.52
GifA11184	AA94599	pottery	black matrix	I	C9040	-	1.5	-22	2570 ± 90	820 BC 693 BC	726 BC 541 BC	0.41 0.59	895 BC 850 BC	871 BC 411 BC	0.02 0.98

Radiocarbon dates were calibrated using Calib Rev 5.0.1 (Reimer *et al.* 2004)

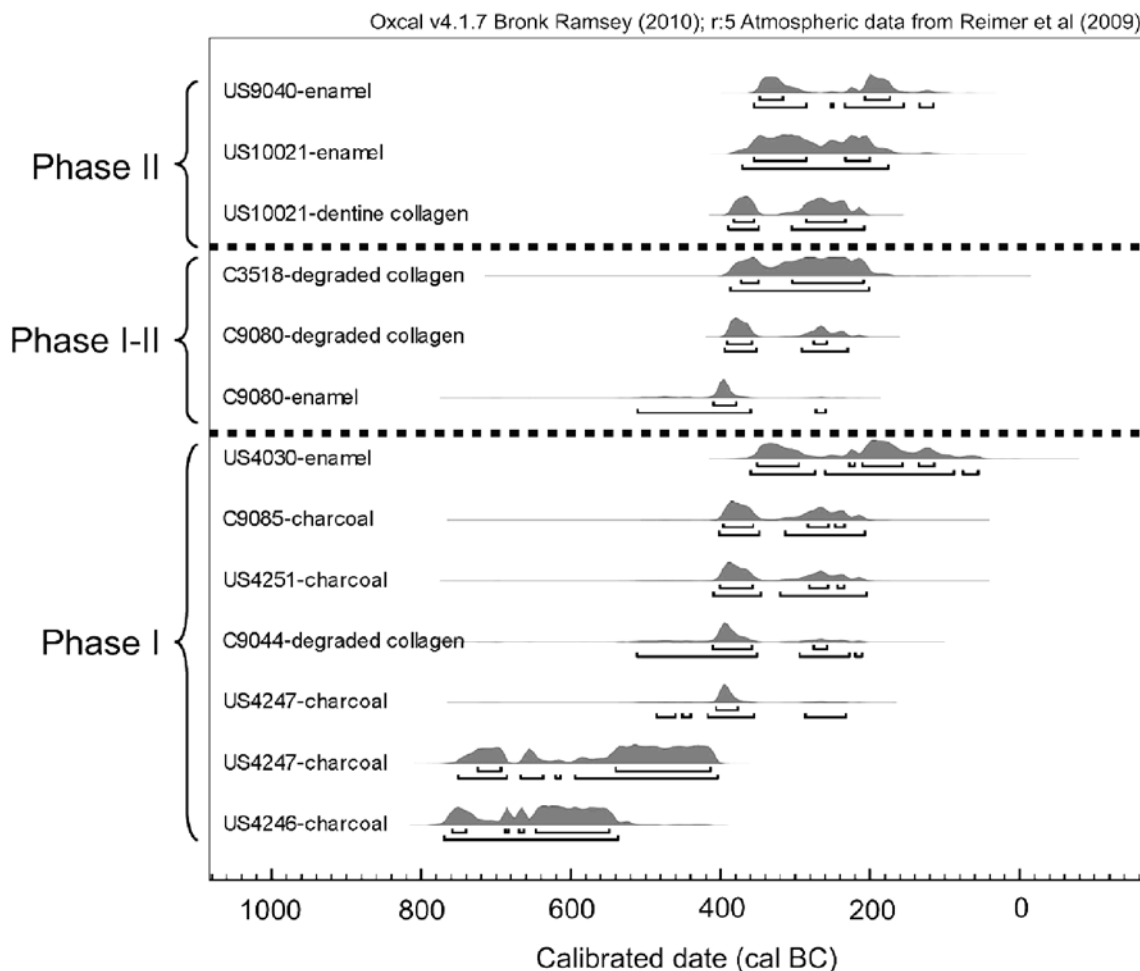


Fig. 1: Radiocarbon results (uncalibrated) obtained in the Hellenistic levels (after Renel *et al.* 2012).

is of no interest because of the existence of a wide plateau in the calibration curve. The five other dates are associated with a cluster of dates around the first half of the 4th century BC, with a high probability (five > 0.6 for 1 σ , of which three > 0.8, 1 σ). This group provides a good evaluation for the Hellenistic phase I.

Phases I-II

This intermediate phase is dated using an ovicaprine tooth and two burnt bones. After calibration, the tooth enamel (C9080) returned a very precise date (table II) (408–383 BC, 1 σ). The burnt bone comes from the same sounding (C9080) and gave a date between 392 and 259 BC (1 σ), but with a high probability (> 0.77) of being between 392 and 359 BC (1 σ). The second burnt bone (C3518) suggests a probable existence sometime between 373 and 209 BC (1 σ) with a high probability (> 0.8) of being between 304 and 209 BC, which brings this date close to phase II.

Phase II

This phase was dated using two ovicaprine teeth (table II, fig. 1). On one tooth (US 10021), two measurements, one from the enamel and the other from the collagen in the dentine, produced dates very close together (2194 \pm 32 ¹⁴C yr BP and 2247 \pm 21 ¹⁴C yr BP, respectively). In spite of the quality of the measurements, the calibration introduces a large uncertainty. The date of the enamel is between 356 and 200 BC (1 σ) and that of the collagen from the dentine between 382 and 234 BC (1 σ), with no possibility of reducing this gap by playing on the probabilities. The second tooth, although dated with great precision (2162 \pm 21 ¹⁴C yr BP), gave a large calibrated interval between 348 and 174 BC (1 σ). This result for phase II covers two centuries, from the first half of the 4th century to the first half of the 2nd century BC. Due to the limited number of samples dated and the presence of large fluctuations in the calibration curve during this period, it is not possible to be more precise.

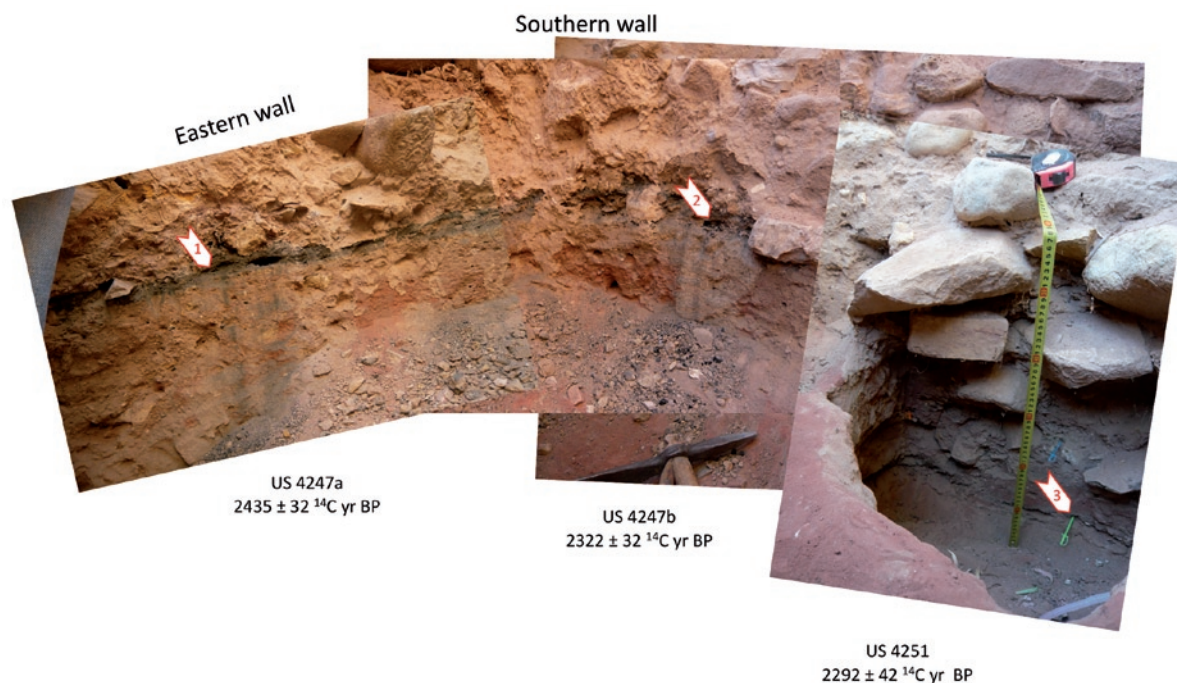


Fig. 2: Picture showing the ashy layer in US 4247a and b and the corresponding ^{14}C dates. Arrows indicate the location of the sampling for ^{14}C dating.

TEST DATING OF THE POTTERY

As a supplement to this study, we took advantage of the opportunity offered by the ANR to try test dating two sherds. The majority of Roman, Greek and Nabataean pottery does not lend itself to dating by ^{14}C , with the exception of a few poorly fired vessels which might have preserved a significant quantity of organic residues within the sherd. An alternative solution lies in the study of carbon residues in the bottom of a vessel, from food remains or organic slips. As an example, Rutgers *et al.* (2007) dated the soot from lamps from the catacombs in Rome. In Petra, we sorted many sherds from Hellenistic levels before finding two which seemed interesting (C11017, C9040). Sherd from C11017, which can be described as coarseware (fig. 3), seems to have been poorly fired, which is confirmed by a high percentage of carbon (1.1%, $\delta^{13}\text{C} = -20.2\text{‰}$) within the sherd. Given that a large part of the organic matter is burnt during firing, this remaining high percentage is a good indicator that organic residues (plants, animal dung?) were added to the clay paste, supplementing the carbon content of the paste. The accuracy of the date is determined by the ratio between temper carbon and clay carbon. After pre-treatment, the date from the inside of the sherd was 2288 ± 47 ^{14}C yr BP (401–235 BC), which is perfectly compatible with the archaeological estimates. On this same

sherd, we dated the organic matter impregnated in the surface (2.8% carbon). The $\delta^{13}\text{C}$ value (-11.3‰) was radically different and indicative of C4 grasses and the age (1595 ± 44 ^{14}C yr BP) indicated that some grass roots were stuck onto the sherd; their date has nothing to do with the age of the pottery. Pollution could also explain the young date (1750 ± 60 ^{14}C yr BP) obtained on a second aliquot taken within the sherd. The second sherd (fig. 3) was fairly similar to the first one. In section, it showed a black part which we dated at 2570 ± 90 ^{14}C yr BP. Both the carbon content (1.5%) and the $\delta^{13}\text{C}$ value (-22.0‰) were similar to that of the previous sherd. Although the large uncertainty of the measurement (± 90 yr) affects the precision of the calibrated date (820–541 BC, 1σ), this result suggests that the sherd could predate the Hellenistic period.

DISCUSSION AND CONCLUSION

From the point of view of ^{14}C methodology, the use of the various available materials proved to be useful. Of course, each material type has its drawbacks. For teeth (US 4030), small traces of secondary carbonates might have slightly decreased the age of the enamel and some charcoal might have come from old wood (US 4246 and 4247a). This result confirms the idea that sampling in arid areas should, in preference,

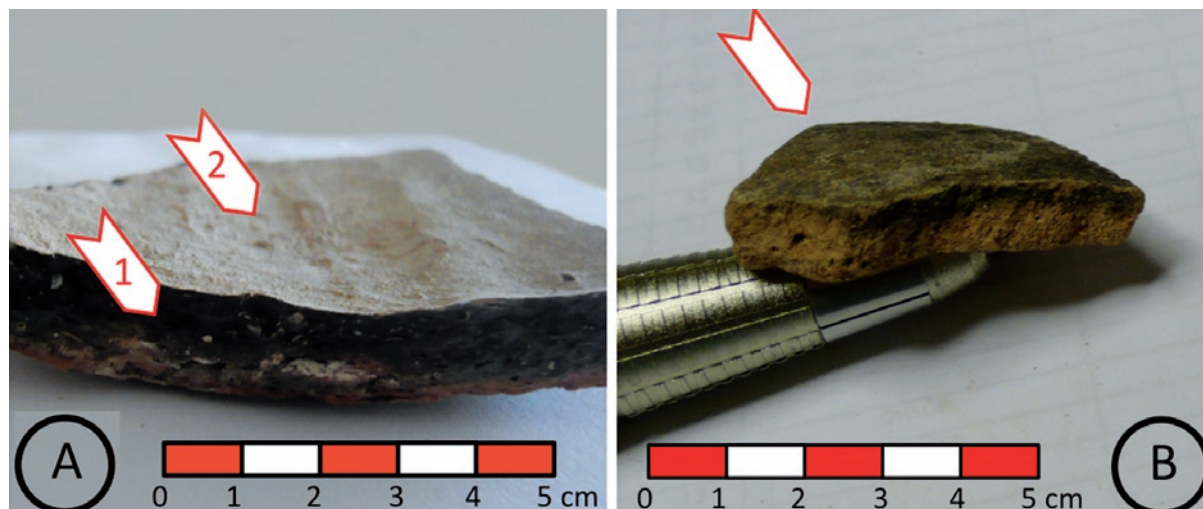


Fig. 3: Picture from the dated sherds coming from levels C11017 (A) and C9040 (B). Arrows indicate the location of the sampling for ^{14}C dating.

use bones and teeth (bioapatite), at least for the recent Holocene. However, it is necessary to continue to date charcoal and, if possible, to look for samples from short-lived sources (e.g. Bourke *et al.* 2007). For certain periods, the calibration curve is very irregular, sometimes causing some large areas of uncertainty. This is particularly the case around 2400–2500 ^{14}C yr BP, and to a lesser extent around 2200–2100 yr BP (uncalibrated). These uncertainties are often of the same order, or occasionally greater than those of the ceramic and

coin typologies. This insurmountable limitation is an integral part of the ^{14}C method and has led certain archaeologists to doubt the usefulness of ^{14}C for these periods (Demoule 1995). But, advances in AMS technology have highlighted other limitations linked to stratigraphy and the fixing of reference typologies (circulation of pottery and coins). The two sides, geochemical and archaeological, must declare their limitations clearly before it can be decided whether or not the combination of their different approaches can

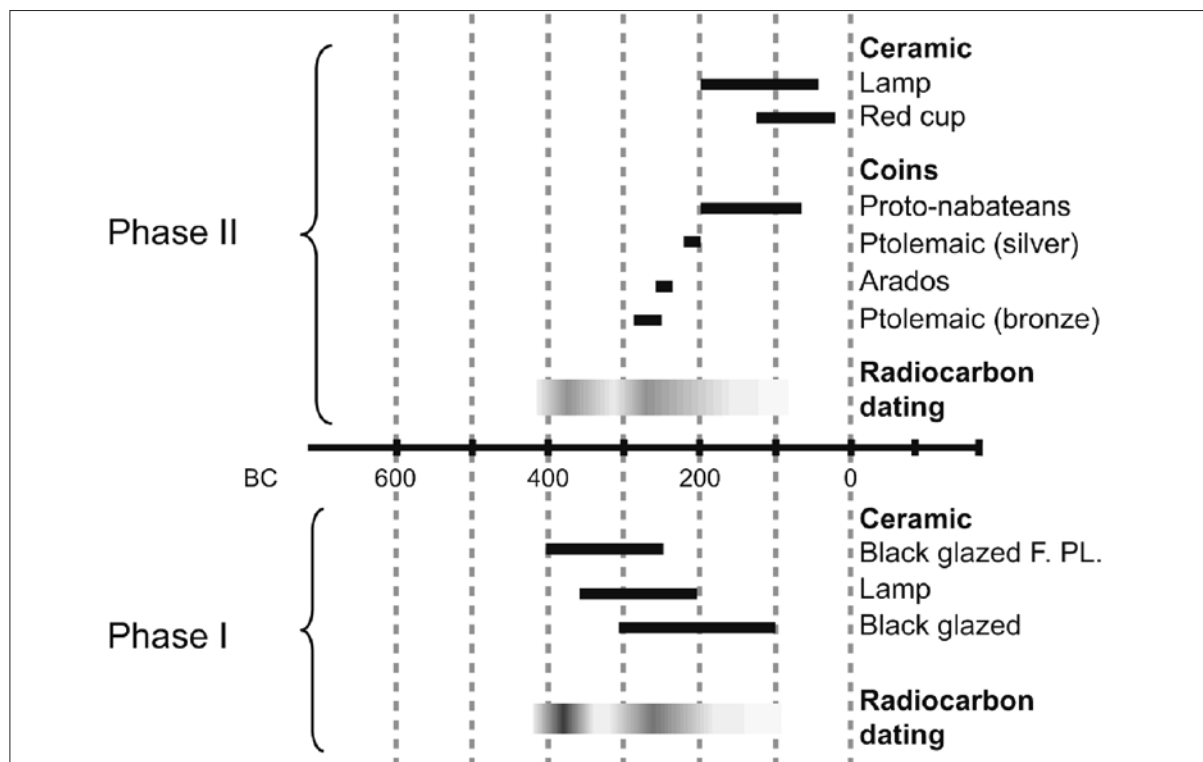


Fig. 4: Comparison between the range of dates provided by the radiocarbon analysis and the diagnostic artefacts (after Renel *et al.* 2012).

refine the chronology. It is in this positive spirit that the chronology of the Hellenistic levels of the urban site of Petra has been developed (Renel *et al.* 2012).

Most of the samples push back the oldest phase, phase I, to between the early 4th and the 3rd century BC. Six samples have a strong probability (>0.6 , 1σ , even >0.8 , 1σ for three of them) of dating to the first half of the 4th century BC. Nevertheless, two charcoal samples (US 4246 and US 4247) indicate an earlier date, between the middle of the 8th and the end of the 5th centuries BC. The sample from US 4246, collected from under a stone in the lowest archaeological level, could belong to an older deposit preserved on the surface of virgin soil. The sample from US 4247 comes from an ashy layer which could contain remains of large timber that was felled when already old. We note, however, that the highest probability (0.81, 1σ) for the charcoal from the ash layer is between 541 and 413 cal BC, not too far from the cluster of dates around the beginning of the 4th century BC. Apart from the two charcoal pieces and one enamel sample (US 4030), this interval is represented in the majority of the samples.

Phase II was dated using two ovicaprine teeth, from one of which two measurements were obtained, using tooth enamel and dentine collagen. The two samples gave essentially the same date (2194 ± 32 ^{14}C yr BP and 2247 ± 21 ^{14}C yr BP, respectively). Despite the low error bars associated with these measurements, calibrated dates are widely-spaced

and fall between 356–200 BC and 382–234 BC, 1σ . The second tooth was also dated with great precision (2162 ± 21 ^{14}C yr BP) but returned a large calibrated range (348–174 BC, 1σ).

On the one hand, the correlation in dating information between the archaeological material and the radiocarbon dates confirms the radiocarbon results (fig. 4). On the other hand, the ^{14}C dates provide the necessary archaeometric confirmation of the archaeological data. Thus, it seems sure that phase I is earlier than the 3rd century BC, the highest probability being that this phase goes back to the 4th century BC. The construction of dwellings in phase II must be dated to the 3rd century BC, their occupation continuing into the 2nd and 1st centuries BC.

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References

- AITKEN M. J. 1985. *Thermoluminescence dating*, London, Academic Press Editions.
- AL-BASHAIREH K. & HODGINS G. 2011. AMS ^{14}C dating of organic inclusions of plaster and mortar from different structures at Petra-Jordan, *Journal of Archaeological Science* 38: 485-491.
- ATLEY S. P. DE 1980. Radiocarbon dating of ceramic materials: progress and prospects. *Radiocarbon* 22(3): 987-996.
- BERGER R. 1992. Carbon14 dating mortar in Ireland, *Radiocarbon* 34: 880-889.
- BERSTAN R., STOTT A.W., MINNITT S., BRONK RAMSEY C., HEDGES R.E.M. & EVERSHERD R.P. 2008. Direct dating of pottery from its organic residues: new precision using compound-specific carbon isotopes, *Antiquity* 83: 702–713.

- BOCHERENS H. 1992. *Biogéochimie isotopique (^{13}C , ^{18}O) et paléontologie des vertébrés : application à l'étude des réseaux trophiques révolus et des paléoenvironnements*, Ph-D thesis, University Paris VI, Paris : 317 p.
- BOURKE S., ZOPPI U., MEADOWS J., HUA Q. & GIBBINS S. 2009. The beginning of the Early Bronze Age in the north Jordan Valley: new ^{14}C determinations from Pella in Jordan, *Radiocarbon* 51(3): 905–913.
- BRONK RAMSEY C. 1995. Radiocarbon calibration and analysis of stratigraphy: the OxCal program, *Radiocarbon* 37 (2): 425–430.
- BRONK RAMSEY C., HIGHAM T & LEACH P. 2004. Towards high-precision AMS: progress and limitations, *Radiocarbon* 46(1):17–24.
- BRONK RAMSEY C., DEE M.W., ROWLAND J., HIGHAM T., HARRIS S., BROCK F., QUILÈS A., WILD E., MARCUS E. & SHORTLAND A. 2010. Radiocarbon - Based Chronology for Dynastic Egypt, *Science* 328: 1554-1557.
- CARAN S.C., WINSBOROUGH B.M., VALASTRO S., NEELY J.A. & ANDERSON R.S. 2001. Radiocarbon chronology of carbonate deposits; reliable dating using organic residues, *Geological Society of America* 33(6): 254.
- CONSTANTIN C. & COURTOIS L. 1985. *Le matériau céramique comme caractéristique culturelle. L'exemple du dégraissant pendant le Néolithique dans le bassin parisien*, Documents et travaux de l'Institut Géologique Albert de Lapparent 9: 19-26.
- CONSTANTIN C. & KUIJPER W. 2002. Utilisation de mousse comme dégraissant dans des céramiques néolithiques de France et de Belgique, *Bulletin de la Société Préhistorique Française* 99: 775-783.
- DELQUÉ-KOLIC E. 1995. *Méthodes d'extraction du carbone des poteries pour leur datation par le radiocarbone*. PhD. thesis, Lyon.
- DEMOULE J.-P. 1995. Les datations au carbone 14 sont-elles utiles ? in *Actes du XXe colloque inter-régional sur le Néolithique, Evreux, oct. 1993*, Revue Archéologique de l'Ouest 7: 135-138.
- FOLK R.L. & VALASTRO S. JR 1976. Successful technique for dating of lime mortar by carbon-14, *Journal of Field Archaeology* 3: 203-208.
- GABASIO M., EVIN J., ARNAL G. B. & ANDRIEUX, P. 1986. Origins of carbon in potsherds, *Radiocarbon* 28 (2A): 711-718.
- HATTÉ C., SALIÈGE J.-F., SENASSON D. & BOCOUM H. 2010. Cultural and trade practices in Sincu Bara (Senegal): a multi-proxy investigation, *Journal of Archaeological Science* 37: 561–568.
- HEDGES R.E.M., CHEN T. & HOWSLEY R.A. 1992. Results and methods in radiocarbon dating of pottery, *Radiocarbon* 34 (3): 906-915.
- HEDGES R.E.M. & MILLARD A.R. 1995. Measurements and relationships of diagenetic alteration of bone from three archaeological sites, *Journal of Archaeological Science* 22: 201-209.
- HEINEMEIER J., RINGBOM Å., LINDROOS A. & SVEINBJÖRNSDÓTTIR Á. 2010. Successful AMS ^{14}C dating of non-hydraulic lime mortars from the medieval churches of the Åland islands, Finland, *Radiocarbon* 52 (1): 171–204.
- HIGHAM T.F.G., JACOBI R., BASELL L., BRONK RAMSEY C., CHIOTTI L. & NESPOULET R. 2011. Precision dating of the Palaeolithic: A new radiocarbon chronology for the Abri Pataud (France), a key Aurignacian sequence, *Journal of Human Evolution* 61(5): 549-563.
- JOHNSON R.A., STIPP J.J., TAMERS M.A., BONANI G., SUTER M. & WOLFLI W. 1986. Archaeologic sherd dating: Comparison of thermoluminescence dates with radiocarbon dates by beta counting and Accelerator techniques, *Radiocarbon* 28(2A): 719-725.
- JULL A.J.T., BURR G.S., MC HARGUE L.R., LANGE T.E., LIFTON N.A., BECK J.W., DONAHUE ? & LAL D. 2004. New frontiers in dating of geological, paleoclimatic and anthropological application using accelerator mass spectrometry measurements of ^{14}C and ^{10}Be in diverse samples, *Global and Planetary Change* 41: 309-323.
- LANTING J.N., AERTS-BIJMA A.T. & VAN DER PLICHT J. 2001. Dating cremated bone, *Radiocarbon* 43: 249–254.
- LONGIN R. 1971. New method of collagen extraction for radiocarbon dating, *Nature* 230: 241-242.

- PIPERNO D.R. & STOTHERT K.E. 2003. Phytolith evidence for Early Holocene Cucurbita domestication in southwest Ecuador, *Science* 299: 1054–1057.
- RALPH E.K. 1959. University of Pennsylvania radiocarbon dates, 3, *American Journal Science, Radiocarbon Supplement* 1: 45-50.
- RECH J.A., FISCHER A.A., EDWARDS D.R. & JULL A.J.T. 2003. Direct dating of plasters and mortars using AMS radiocarbon: A pilot project from Khirbat Qana, Israel, *Antiquity* 77: 155-174.
- REIMER P.J., BAILLIE M.G.L., BARD E., BAYLISS A., BECK J.W., BERTRAND C.J.H., BLACKWELL P.G., BUCK C.E., BURR G.S., CUTLER K.B., DAMON P.E., EDWARDS R.L., FAIRBANKS R.G., FRIEDRICH M., GUILDERSON T.P., HOGG A.G., HUGHEN K.A., KROMER B., MCCORMAC G., MANNING S., RAMSEY C.B., REIMER R.W., REMMELE S., SOUTHON J.R., STUIVER M., TALAMO S., TAYLOR F.W., VAN DER PLICHT J. & WEYHENMEYER C.E. 2004. IntCal04 terrestrial radiocarbon age calibration, 0-26 cal kyr BP, *Radiocarbon* 46: 1029-1058.
- RENEL F., MOUTON M., AUGÉ CH., GAUTHIER C., HATTÉ CH., SALIÈGE J.-F. & ZAZZO A. 2012. Dating the early phases under the temenos of the Qasr al-Bint at Petra, *Proceedings of the Seminar for Arabian Studies* 42 (in press).
- RUTGERS L., VAN DER BORG K. & DE JONG A. 2007. Radiocarbon dating of several ancient Jewish oil lamps from Rome, *Radiocarbon* 49(3): 1215–1219.
- SALIÈGE J.-F. & PERSON A. 1991. Matière organique des céramiques archéologiques et datation par la méthode du carbone 14, in Raimbault M., Sanogo K. (eds.) *Recherches archéologiques au Mali*, Paris, Karthala Editions: 413-448.
- SALIÈGE J.-F., PERSON A. & PARIS F. 1995. Preservation of $^{13}\text{C}/^{12}\text{C}$ original ratio and ^{14}C dating of the mineral fraction of human bones from Saharan tombs, Niger, *Journal of Archaeological Science* 22: 301-312.
- SALIÈGE J.-F. & PESSIN H. 2005. Datation ^{14}C de Tell Shiukh Fawqani : la méthode et ses limites, in Bachelot L., Fales F. M. (eds.) *History of the Ancient Near East*, Padova, S.A.R.G.O.N. Editions: 1075-1079.
- SERENO P., GARCEA E., JOUSSE H., STOJANOWSKI C., SALIÈGE J.-F., MAGA A., IDE O., KNUDSON K., MERCURI A.-M., STAFFORD T., KAYE T., GIRAUDI C., MASSAMBA N'SIALA I., COCCA E., MOOTS H., DUTHEIL D. & STIVERS J. 2008. Lakeside cemeteries in the Sahara: 5000 years of Holocene population and environmental Change, *PLoS ONE* 3(8): 2995.
- SMITH B. 1997. Reconsidering the Ocampo caves and the era of incipient cultivation in Mesoamerica, *Latin American Antiquity* 8(4): 342–83.
- STOTT A.W., BERSTAN R., EVERSHERD R.P., BRONK RAMSEY C., HEDGES R.E.M. & HUMM M.J. 2003. Direct dating of archaeological pottery by compound-specific ^{14}C analysis of preserved lipids, *Analytical Chemistry* 75: 5037–5045.
- STUCKENRATH J. 1963. University of Pennsylvania radiocarbon dates 6, *Radiocarbon* 5: 82-103.
- WRIGHT L.E. & SCHWARCZ H.P. 1996. Infrared and isotopic evidence for diagenesis of bone apatite at Dos Pilas, Guatemala: palaeodietary implications, *Journal of Archeological Science* 23: 933-944.
- ZAZZO A., LÉCUYER C. & MARIOTTI A. 2004. Experimentally-controlled carbon and oxygen isotope exchange between bioapatites and water under inorganic and microbially-mediated conditions, *Geochimica et Cosmochimica Acta* 68: 1-12.
- ZAZZO A. & SALIÈGE J.F. 2011. Radiocarbon dating of biological apatites: a review, *Palaeogeography, Palaeoclimatology, Palaeoecology* 310: 52-61.
- ZAZZO A., SALIÈGE J.-F., LEBON M., LEPETZ S. & MOREAU C. 2012. Radiocarbon dating of calcined bones: insights from combustion experiments under natural conditions, *Radiocarbon* (in press).
- ZOURIDAKIS N., SALIÈGE J.-F., PERSON A. & FILIPPAKIS S. 1987. Radiocarbon dating of mortars from ancient Greek palaces, *Archaeometry* 29 (1): 60-68.

