

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

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

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

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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)



Berlin 2013

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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 ¹⁴C 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 ¹⁴C 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 ¹⁴C 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 (14C). One part of the French-German ANR funded "Early Petra" project consisted of comparing the ¹⁴C 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 ¹⁴C 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 ¹⁴C 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 ¹⁴C 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 ¹⁴C 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 ¹⁴C activity of the first ring gives the time elapsed since the beginning of the tree life, whereas ¹⁴C 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 ¹⁴C 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. ¹⁴C 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 ¹⁴C dates from human bioapatite was tested successfully during the excavations of the large necropolises of Aïr 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 °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 ¹⁴C (Lanting et al. 2001). Above 600°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 CO₂ and / or CO₂ 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 CO₂ 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 ¹⁴C 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 though 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 protohistoric 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 semiarid 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 (Sereno 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 pretreatment 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 μ m. Then the secondary carbonates were eliminated using acid in a vacuum (1M CH₃COOH). The CO₂ was extracted using phosphoric acid in vacuum, at 70°C.

• Degraded collagen underwent an AAA pretreatment 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 CO_2 was extracted by combustion at 800°C in the presence of O_2 .

• 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 ${}^{14}C / {}^{12}C$ 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 \pm 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, 1 σ) 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, 1 σ) and 1868 ± 34 ¹⁴C yr BP (AD 84–210, 1 σ), and for the upper level 35–40 cm above the pavement, a date of 1877 ± 75 ¹⁴C yr BP (AD 61–231, 1 σ). 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, 10), 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, 10) 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 $(0.81, 1\sigma)$ 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±39¹⁴C 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

levels
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Table

										68.2%			95.4%	
prep	AMS	description	fraction	phase	SU	$\delta^{I3}C$ %0	14 C âgu	BP	from	to	proba	from	to	proba
Muse 14	AA90694	ovi-caprinae	enamel	Nabataean	C 4178	-7.3	1904	E 31	68 AD	128AD	1.00	25 AD	43 AD	0.04
												46 AD	178 AD	0.92
												190 AD	212 AD	0.04
Muse 15	AA90695	camel	enamel	late Nabataean	C 4178	-5.9	1813	E 30	138 AD	160 AD	0.24	126 AD	259 AD	0.95
									165 AD	197 AD	0.36	295 AD	322 AD	0.05
									207 AD	239 AD	0.40			
GifA- 10 272	AA88862	charcoal	ı	Roman	C 3110, 8 cm	-22.8	1873	E 59	77 AD	214 AD	1.00	3 AD	258 AD	0.98
												298 AD	320 AD	0.02
GifA- 10 273	AA88863	charcoal	ı	Roman	C 3110, 8 cm	-23.2	1868	г 34	84 AD	143 AD	0.62	72 AD	232 AD	1.00
									147 AD	171 AD	0.21			
									193 AD	210 AD	0.16			
GifA- 10 274	AA88864	charcoal	ı	Roman	C 3108, 35-40 cm	-22.8	1877	E 75	61 AD	231 AD	1.00	41 BC	263 AD	0.94
												277 AD	330 AD	0.06
Muse 16	AA90696	camel	enamel	Roman	C 4116	-8.4	1782	E 20	219 AD	258 AD	0.69	139 AD	196 AD	0.14
									297 AD	321 AD	0.31	208 AD	262 AD	0.54
												278 AD	329 AD	0.32
Muse 17	AA90697	ovi-caprinae	enamel	late Roman	C 2006 / 2000	-4.9	2419	г 33	697 BC	696 BC	0.01	748 BC	687 BC	0.17
									538 BC	407 BC	0.99	666 BC	643 BC	0.04
												591 BC	<i>577</i> BC	0.01
												567 BC	400 BC	0.77
966d	AA90699	human tooth	collagen	Abbassid	grave 12	-22.9	1180	E 120	711 AD	746 AD	0.13	640 AD	1047 AD	0.98
									766 AD	974 AD	0.87	1089 AD	1122 AD	0.02
												1139 AD	1149 AD	0.01
Radiocarbon dat	es were calibra	ated using Calib Re	ev 5.0.1 (Rei	mer <i>et al</i> . 2004)										

_																	~									
		proba	0.48	0.49	0.02	1.00			0.35	0.65		1.00		0,58	0.41	0.01	0.13	0.85	0.02	0.34	0.64	0.03		0.47	0.53	
0. 10/	95.4%	to	287 BC	157 BC	117 BC	177 BC			350 BC	209 BC		201 BC		353 BC	230 BC	215 BC	434 BC	360 BC	260 BC	275 BC	89 BC	57 BC		349 BC	208 BC	
		from	355 BC	233 BC	134 BC	369 BC			389 BC	304 BC		387 BC		396 BC	292 BC	217 BC	510 BC	428 BC	273 BC	359 BC	260 BC	75 BC		402 BC	315 BC	
		proba	0.47	0.53		0.66	0.03	0.30	0.40	0.42	0.19	0.19	0.81	0.77	0.23		1.00			0.39	0.03	0.46	0.12	0.6	0.29	0.12
100.00	68.2%	to	317 BC	174 BC		285 BC	249 BC	200 BC	356 BC	254 BC	234 BC	350 BC	209 BC	359 BC	259 BC		383 BC			296 BC	221 BC	157 BC	115 BC	357 BC	256 BC	234 BC
	-	from	348 BC	207 BC		356 BC	254 BC	234 BC	382 BC	285 BC	249 BC	373 BC	304 BC	392 BC	275 BC		408 BC			352 BC	228 BC	210 BC	135 BC	397 BC	284 BC	247 BC
	RP	ň	21			32			21			40		21			32			39				39		
	14 C 000	c use	$2162 \pm$			2194 ±			2247 ±			2227 ±		2268 ±			2332 ±			2153 ±				2278 ±		
	813C %	0 ~ 00	-9.3			-8.2			-22.4			-17.3		-15.4			-10.3			-9.1				-21.2		
	J%	201	ı			ı						ı					ı			ı				ı		
	context	COMEN	rich organic layer			filling of a pit			filling of a pit			wall foundation		wall foundation			wall foundation			on the virgin soil				terrace wall		
	SII	20	US9040			US 10021			US 10021			C3518		C 9080			C 9080			US 4030				C9085		
	ospyu	pennd	Π			п			Π			II-II		II-II			II-II			П				-		
	fraction	Jucaon	enamel			enamel			dentin collagen			decomposed collagen		decomposed collagen			enamel			enamel				ı		
	descrintion	unindi incon	ovi-caprinae			ovi-caprinae			ovi-caprinae			burnt bone		burnt bone			ovi-caprinae			ovi-caprine				charcoal		
	SWF	CINIC	AA90688			AA90689			AA90690			AA92554		AA90693			AA90691			AA92552				AA88859		
	nren	prep	Muse 11			Muse 12			866d			P1041		P988			Muse 13			P1039				GifA-10269		

Table II : Radiocarbon dates obtained in the Hellenistic levels

0.54 0.46	0.10 0.71 0.18 0.01	0.02 0.01 0.88 0.08	0.22 0.07 0.01 0.70	0.23		0.48 0.52	0.02 0.98
347 BC 207 BC	434 BC 352 BC 228 BC 211 BC	460 BC 440 BC 356 BC 233 BC	686 BC 638 BC 614 BC 405 BC	698 BC 538 BC		337 BC 203 BC	871 BC 411 BC
409 BC 319 BC	510 BC 428 BC 295 BC 220 BC	486 BC 451 BC 418 BC 287 BC	751 BC 667 BC 621 BC 595 BC	769 BC 696 BC		410 BC 330 BC	895 BC 850 BC
0.69 0.24 0.07	0.92 0.08	1.00	0.18 0.01 0.81	0.13 0.04 0.05 0.77		0.58 0.29 0.12	0.41 0.59
357 BC 257 BC 235 BC	360 BC 261 BC	381 BC	694 BC 656 BC 413 BC	742 BC 683 BC 663 BC 550 BC		356 BC 253 BC 234 BC	726 BC 541 BC
401 BC 282 BC 244 BC	410 BC 273 BC	405 BC	724 BC 656 BC 541 BC	759 BC 689 BC 670 BC 647 BC		401 BC 285 BC 250BC	820 BC 693 BC
2292 ± 42	2318 ± 40	2322 ± 32	2435 ± 32	2495 ± 22	1595 ± 44 1750 ± 60	2288 ± 47	2570 ± 90
-19.4	-20.3	-21	-21.4	-22.8	-11.3	-21	-22
I	I	I	1	1	2.8 1.1		1.5
pit with virgin soil	on the virgin soil	ashy layer	ashy layer	ashy layer			
US 4251	US 9044	US 4247b	US 4247a	US 4246	US 11017 US 11017	US 11017	C9040
П	ц	Ι	Ι	Ι	I		
1	decomposed collagen		1	1	surface black matrix	black matrix	black matrix
charcoal	burnt bone	charcoal	charcoal	charcoal	pottery pottery	pottery	pottery
AA88861	AA92553	AA88857	AA88856	AA88855	AA88865 AA94595	AA88866	AA94599
GifA-10271	P1040	GifA-10267	GifA-10266	GifA-10265	GifA-10276 GifA-11182	GifA-10277	GifA11184

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



Oxcal v4.1.7 Bronk Ramsey (2010); r:5 Atmospheric data from Reimer et al (2009)

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±32 ¹⁴C yr BP and 2247±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 (1σ) 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±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 ¹⁴C dates. Arrows indicate the location of the sampling for ¹⁴C 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 ¹⁴C, 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%) δ^{13} C= -20.2‰) 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 ¹⁴C 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 δ^{13} C value (-11.3‰) was radically different and indicative of C4 grasses and the age (1595±44 ¹⁴C 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 ¹⁴C 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 ¹⁴C yr BP. Both the carbon content (1.5%) and the δ^{13} 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 suggest that the sherd could predate the Hellenistic period.

DISCUSSION AND CONCLUSION

From the point of view of ¹⁴C 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 ¹⁴C 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 ¹⁴C 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 ¹⁴C method and has led certain archaeologists to doubt the usefulness of ¹⁴C 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, 1 σ) 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 ¹⁴C yr BP and 2247±21 ¹⁴C 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, 1 σ . The second tooth was also dated with great precision (2162±21 ¹⁴C yr BP) but returned a large calibrated range (348–174 BC, 1 σ).

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 ¹⁴C 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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