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Direct ^{14}C-dating of Roman and Late Antique Purple Dye Sites by Murex Shells

Datation directe par le radiocarbone de coquillages murex provenant de teintureries de l'Antiquité romaine et tardive

Mark Van Strydonck*, Mathieu Boudin* and Damià Ramis**

Abstract: Until late antique times, murex shells were used for the production of true purple. Murex production sites are found all around the Mediterranean. In this paper are studied four sites from the Balearic Islands. Radiocarbon dates from animal bones and charcoal supposed to be synchronic with the murex dye production, are compared to direct dating of the murex shells. In all but one case the terrestrial samples were inconsistent. The murex shells on the other hand yielded coherent results. The charcoal and the animal bones were without any doubt intrusive. Consequently the ideal radiocarbon sampling strategy should exhibit a good equilibrium between sample quality and sample integrity.

Résumé : *Jusqu'à la fin de l'Antiquité, les coquilles de murex étaient utilisées pour la production de la véritable pourpre. Des sites de production de murex ont été découverts sur tout le pourtour méditerranéen. Le présent article étudie quatre sites des Iles Baléares. Les datations radiocarbones d'os d'animaux et de charbon de bois, censés être concomitants de la production de teinture à partir du murex, sont comparées avec les datations directes des coquilles de murex. Sauf dans un cas, les échantillons terrestres étaient contradictoires. D'un autre côté, les coquilles de murex fournissaient des résultats cohérents. Le charbon de bois et les os d'animaux étaient donc sans nul doute parasites.*
En conséquence, la stratégie d'échantillonnage radiocarbone idéale reposerait sur un bon équilibre entre la qualité de l'échantillon et son intégrité.

Keywords: radiocarbon dating, murex, sample integrity, Balearic islands.

Mots clé : *datation par le radiocarbone, murex, intégrité des échantillons, Îles Baléares.*

1. Introduction

First used by the Phoenicians, murex shells were used for the production of true purple until late antique times (Gleba and Vanden Berghe, in print). Murex production sites are found all around the Mediterranean, and the Balearic Islands form no exception (Costa, 2009). The purple dyestuff is not present in the living animal, but after the animal dies it can develop through enzymatic hydrolysis of uncoloured components. This process has to be initiated immediately after collection of the shells. During this preparation an unpleasant smell is produced (rotting of the animal), hence it was forbidden to perform this activity in urban areas. Consequently purple dye production was done on site. As a result these sites contain very limited archaeological remains, except for large quantities of murex shell debris (Alfaro *et al.*, 2004). In this paper shells, charcoal and bones coming from 4 Balearic sites were analysed by radiocarbon dating in order to date their use. All shells analysed in this study are *Murex trunculus*(figure 1).

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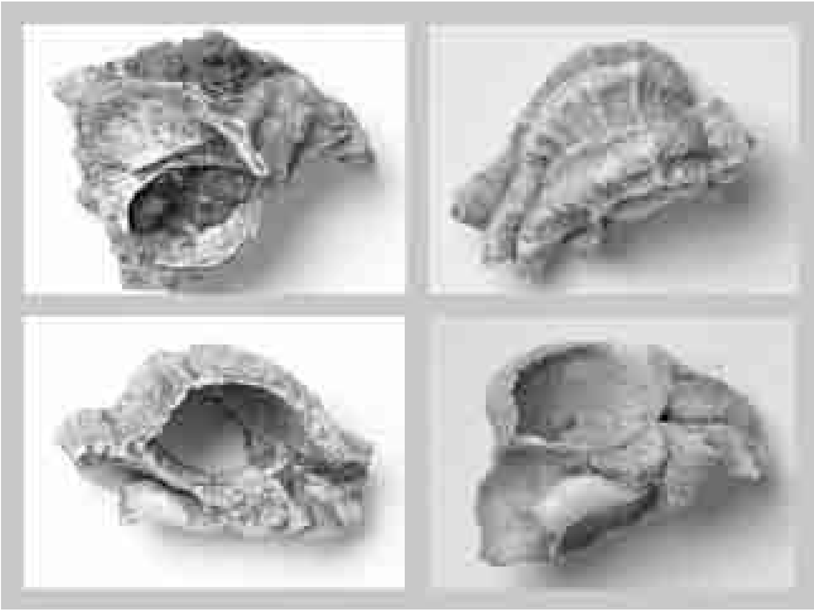

Figure 1: (See colour plate) *Murex trunculus* from purple dye production sites.
Figure 1: (Voir planche couleur) Murex trunculus provenant d'une site de production de la teinture pourpre.

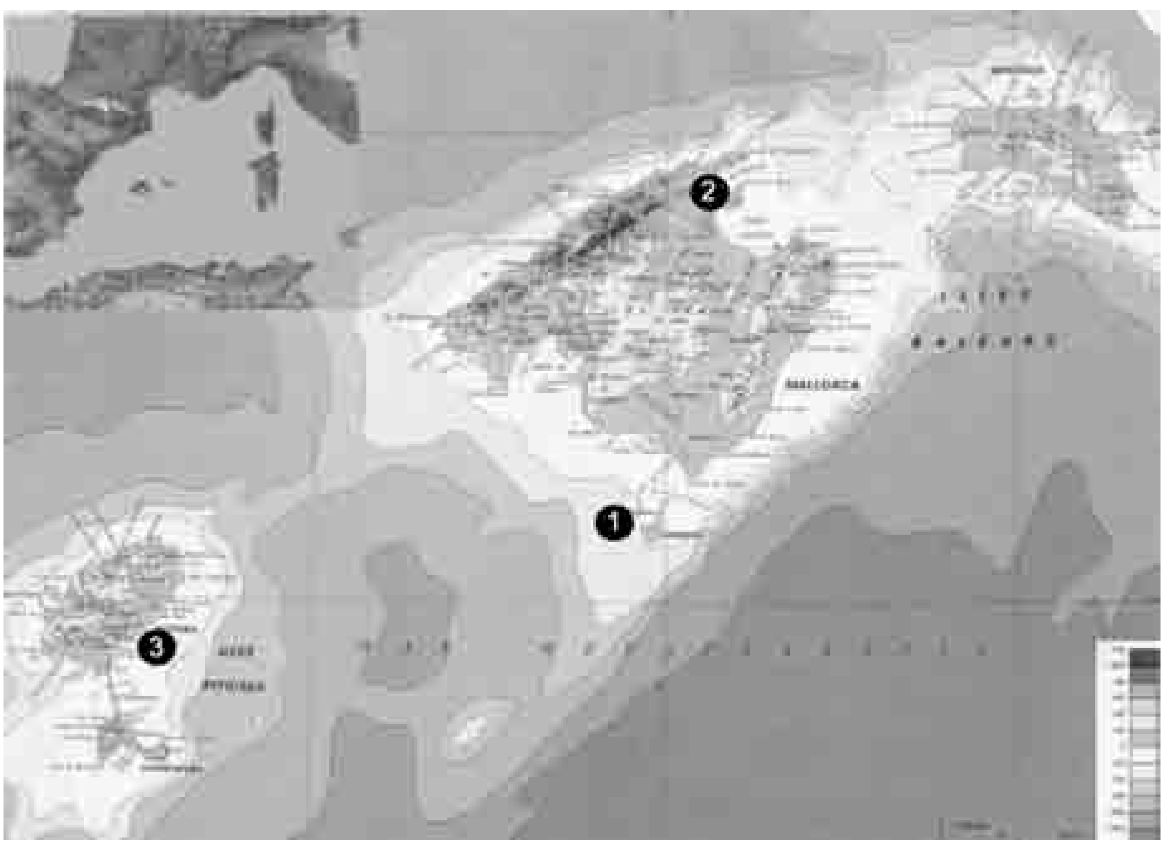

Figure 2: (See colour plate) The Balearic Islands: 1) Cabrera; 2) Pedret de Bóquer; 3) Pou des Lleó – Cala Olivera.
Figure 2 : (Voir planche couleur) Les Îles Baléares : 1) Cabrera ; 2) Pedret de Bóquer ; 3) Pou des Lleó – Cala Olivera.

2. Materials and methods

Sample selection

Mallorca

Cabrera

A letter written by Pope Gregorius Magnum in 603 AD gives unmistakable evidence of the presence of a monastic congregation in the Cabrera Archipelago. Several locations near the port of Cabrera showed evidence of the presence of monks on the archipelago including a necropolis overlaying a purple dye tannery with a high concentration of murex debris (Riera, 2010; Riera & Riera, 2005; Ramis & Pons, 2005). Bone samples from domesticated animals (goat) and murex shells from two locations were analyzed, both supposed to date from the period of the Roman purple dye production (figure 1).

Pedretd eBóqu er

Located just outside the port of Pollença, this is the area where the *Civitas Bocchoritana* was supposedly founded. During the excavation a reservoir lined with mortar was found together with a lot of shells, most of them murex (Estarellas & Merino, 2005). Samples from domesticated animals (bovine and goat) and murex samples, both supposed to date from the period of the purple dye production, were analysed.

Ibiza

Poud esLleó

The site of Canal d'en Martí is located in the Cala del Pou des Lleó, at the north-east coast of Ibiza. It has been recognized as a well-organized area for purple dye production, with a long history. Some architectural remains were recorded: mainly some pits and very eroded walls. The seashell debris related to purple production is very abundant and the characteristic breakage patterns of the specimens are quite common (Alfaro *et al.*, 2002, 2004). In this case charcoal and murex shells were dated, both supposed to date from the period of the purple dye production.

CalaO livera

Cala Olivera is situated near the city of Ibiza, on the east coast of the island. A small collection of seashells – mainly murex – has been excavated there. The archaeological site is interpreted as a small purple dye workshop without any visible architectonic remains (Costa & Alfaro, 2007; Alfaro & Costa, 2008). Also in this case charcoal and murex shells were dated, both supposed to date from the period of the purple dye production.

Sample pre-treatment

Collagen from the bone samples was extracted following the Longin method (Longin, 1971) with an additional NaOH-wash. Collagen quality was tested by C:N (carbon/nitrogen) ratio and collagen yield. A small portion of each collagen sample was transferred in duplicate into tin capsules, which were analyzed using a ThermoFinnigan delta +XL (continuous flow type) isotopic mass spectrometer, interfaced with a Flash EA1112 elemental analyzer via a Conflo III interface. Both elements were measured together and a Helium-dilution was applied for carbon as the amount of C is much greater than the amount of N in the samples. Furthermore the visual appearance of the collagen and the amount of retrieved collagen were considered (Van Strydonck *et al.*, 2005).

Charcoal was successively washed in hot HCl (1%), NaOH (1%) and HCl (1%) (acid-alkali-acid) method and then again washed with Milli-Q water and dried (Van Strydonck *et al.*, 2010).

Shells were cleaned by removing the surface layer of the shell. Sample quality was checked by $\delta^{13}C$ measurements (marine samples have $\delta\ ^{13}C > 0$ ‰) using a Finnigan-Mat- δ isotopic mass spectrometer and the crystal structure was measured by XRD using a Bruker-D8. The XRD measurements are necessary because in the case of marine carbonates, contamination appears usually in the form of secondary low-magnesium calcite – the stable polymorph of calcium carbonate ($CaCO_3$) – and byproduct of the post-mortem recrystallization or replacement of the autochthonous phase, originally in the form of high-magnesium calcite or aragonite. Depending on the nature of the depositional environment, the recrystallized phase may be contemporary in age with the original shell-$CaCO_3$ and may have even derived from it by dissolution-recrystallization reactions, or can be an exogenous contaminant of younger or older age (Magnani *et al.*, 2007).

Graphitization and AMS measurements

CO_2 from the organic samples (charcoal and collagen) was obtained by combustion (in the presence of CuO) and in the case of shells by extraction with phosphoric acid (H_3PO_4).

Graphitisation of CO_2 was carried out using H_2 over a Fe catalyst.

Targets were prepared at the Royal Institute for Cultural Heritage in Brussels (Belgium) (M. Van Strydonck, K. van der Borg 1990-91) and measured at the Leibniz Labor für Altersbestimmung und Isotopenforschung in Kiel (Germany) (Nadeau *et al.*, 1998).

3. Results and discussion

Results

All results are summarized in table 1, the uncalibrated radiocarbon results are depicted in figure 3.

Discussion

Samplequ alityc ontrol

All charcoal samples resisted very well the AAA pre-treatment.

The quality of the bones was also very good. Although the amount of collagen was sometimes low, other parameters such as the C:N ratio, and the general appearance of the collagen were excellent. $\delta^{13}C$ and $\delta^{15}N$ correspond to terrestrial herbivores, no shift due to contamination was observed (Van Strydonck et al. 2005).

All shells show positive $\delta^{13}C$ values, indicating a 100 % marine provenance of the carbon. In some of the samples however calcite was detected. The presence of calcite is a strong indication of the presence of secondary carbonate. This secondary carbonate can have a different ^{14}C signal than the original aragonite from the murex shells. Fortunately our results exclude this possibility. All murex shells from UE501 at Bóquer statistically have the same age although one of the samples contains about 16.2 % calcite. The murex samples from Cabrera statistically have the same age as well, in spite of the presence of different amounts of calcite. This proves that the deposition of secondary carbonate happened shortly after the shells were harvested. This situation is in agreement with the production process of the purple dye.

Samplein tegrity

While the sample quality concerns the reliability of the ^{14}C date, the relation between the samples age and the archaeological phenomenon to be dated is defined by the sample integrity. There should be a close relationship between the radiocarbon age of the material and the human event of interest (Van Strydonck *et al.*, 1999). Only in the case of the Bóquer samples the differences in age between the marine murex shells and the terrestrial bones from domesticated animals are almost constant as one should expect from marine/terrestrial pairs with the same real age (figure 3, table 1). Using the Bóquer samples to calculate the ΔR value (local deviation from the global ocean reservoir age)for the Mediterranean surface waters around the Balearic archipelago, a value of 26±24 ^{14}C year is obtained (Van Strydonck *et al.*, 2010). It should be noted here that the used ΔR value is in agreement with previous research (Siani *et al.*,2000)

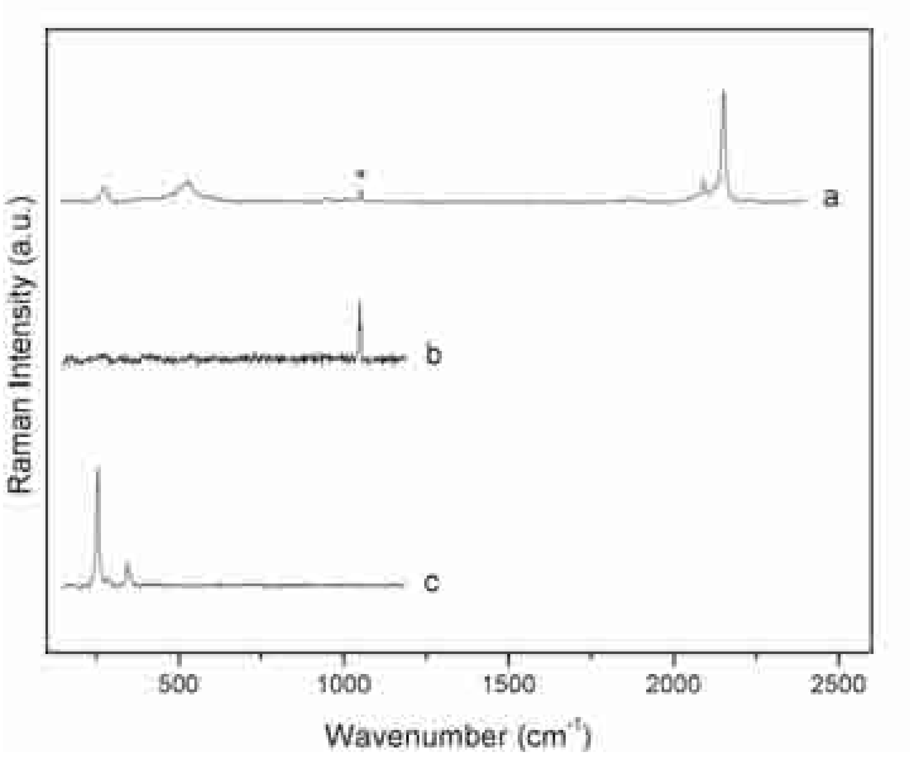

Figure 3: uncalibrated radiocarbon analyses (BP).
Figure 3 : analyses radiocarbone non calibrées (BP).

reference	sample	lab. ref. (KIA-)	BP	± 1σ	% collagen	appearance collagen	δ ^{13}C ‰	δ ^{15}N ‰	C:N	% aragonite	% calcite
Bóquer (Mallorca)											
BO6 UE 401	animal bone	38439	1605	25	3.36	white & fluffy	-20.2	+6.1	3.2		
BO6 UE 501 A	bovine	38442	1705	25	0.79	white & fluffy	-20.0	+4.7	3.2		
BO6 UE 501 B	domesticated animal	38447	1675	25	3.00	white & fluffy	-19.7	+4.9	3.2		
BO6 UE 501 C	horne pit goat	38446	1695	25	3.33	light yellow & fluffy	-19.0	+6.5	3.3		
BO6 UE 501 A	murex	38443	2065	25			+2.4			100.0	
BO6 UE 501 B	murex	38444	2050	30			+2.0			83.8	16.2
BO6 UE 501 C	murex	38445	2070	25			+2.5			100.0	
BO6 UE 401	murex	38440	1980	25			+2.2			100.0	
Cabrera (Mallorca) Test site 1											
PF 03 UE 116-1	goat	38449	1290	25	5.25	white & fluffy	-19.0	+8.4	3.2		
PF 03 UE 116-2	goat	38448	1280	25	1.80	white & fluffy	-19.0	+8.5	3.2		
UE 155-1	murex	38456	1765	25			+0.9			91.7	8.3
UE 155 -2	murex	38441	1825	25			+1.3			87.6	12.4
Cabrera (Mallorca) Test site 2											
PF 05 UE 263	goat	40857	1425	25	6.10	white & fluffy	-19.6	6.2	3.1		
PF 05 UE 265	goat	40858	940	25	8.40	white & fluffy	-19.3	7.0	3.1		
PF 05 UE 305	goat	40859	1285	25	3.50	white & fluffy	-19.7	6.2	3.1		
PF 05 UE 263	murex	40789	1855	20			+0.4			90.9	9.1
PF 05 UE 265	murex	40788	1800	25			+1.1			89.8	10.2
PF 05 UE 305	murex	40790	1840	20			+1.5			85.5	14.5
Pou des Lleó (Ibiza)											
Unit 2204	charcoal	43313	130	30							
Unit 2204	murex	43311	2010	25			+1.8			77.9	22.1
Cala Oliver (Ibiza)											
UE 8 – D1	charcoal	43312	65	25							
UE 8 – C2	murex	43033	2050	30			+0.7			100	

Table 1: Samples and results from dye sites.
Tableau 1 : Échantillons et résultats des sites de teintureries.

In the case of the Cabrera samples important age differences are observed between the terrestrial samples [χ^2 test fails: df=4, T= 204.014 (5%= 9.5)]. The murex samples on the other hand form a much more coherent series.

In the case of the two sites from Ibiza, the charcoal samples are unrealistically young.

Figure 3 depicts the calibrated radiocarbon results (Bronk Ramsey 2001) using the marine data from Reimer et al. (2009) and a $\Delta R = 26 \pm 24$ ^{14}C year for the different sites and units, calculated from the murex dates. From Bóquer UE 501 as well as from Cabrera PF05 the average of 3 results is calculated, from Cabrera 155 the average of 2 results is calculated. A χ^2 test shows that samples originating from the same layer can have the same real age. The results are also summarized in table 2.

The results in table 2 and figure 4 are coherent and correspond to the archaeological expectations. This is not the case for the charcoal samples and the bones from domesticated animals, both yielding dates that are contradictory and too recent.

3. Conclusion

Most archaeologists prefer terrestrial samples to marine samples for dating because the marine reservoir age of large parts of the world is unknown and because of the possibility of contamination. In many cases however sample integrity should be a much more decisive factor than sample quality. There is a much more direct relationship between

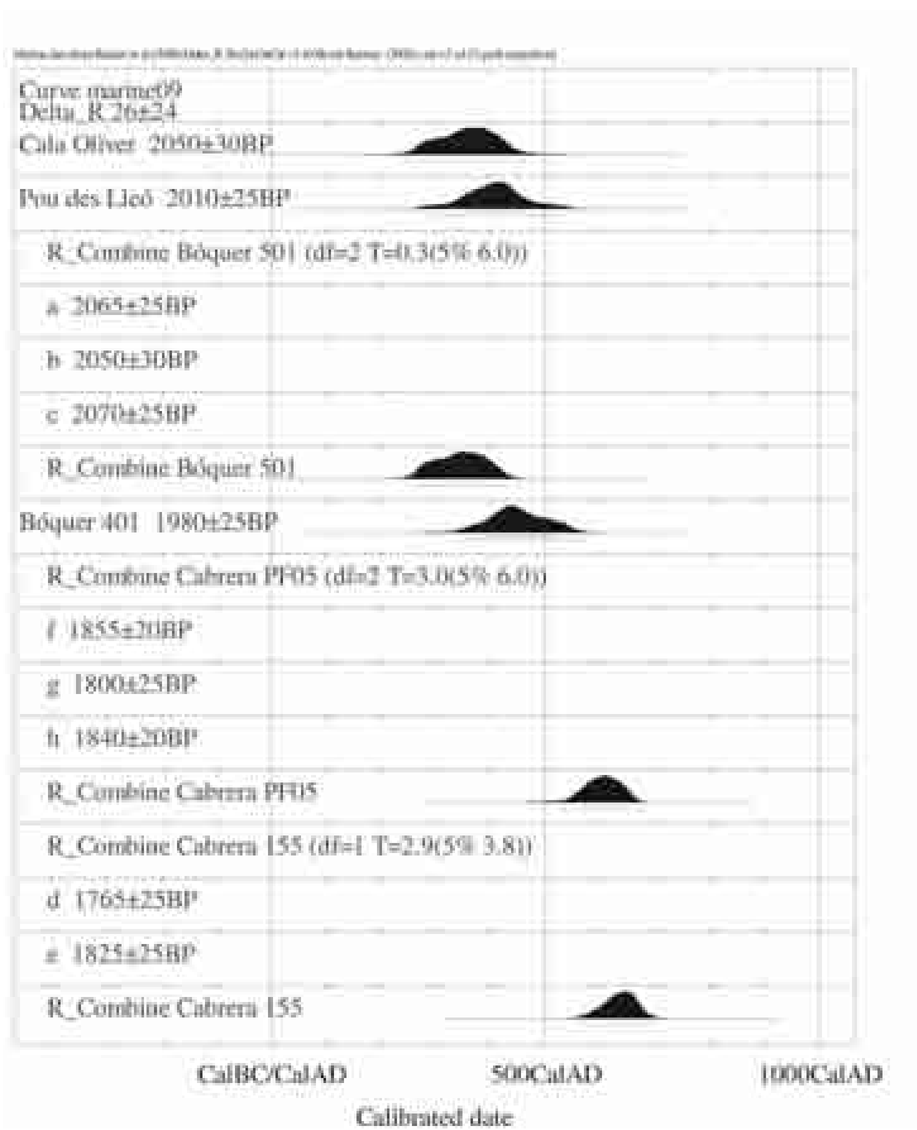

Figure 4: Calibrated radiocarbon dates from murex samples.
Figure 4 : Résultats radiocarbone calibrés des échantillons de murex.

CalaO liver:2050±30BP 300AD (68.2%) 430AD 240AD (95.4%) 460AD	Bóquer401:1980±25BP 390AD (68.2%) 510AD 340AD (95.4%) 560AD
Poud esLleó:2010±25BP 350AD (68.2%) 460AD 290AD(95.4%)530A D	CabreraPF05:1836±12BP 570AD (68.2%) 645AD 530AD (95.4%) 680AD χ2 -Test:d f=2 T=3.0(5%6. 0)
Bóquer501:2063±15BP 290AD (68.2%) 400AD 250AD (95.4%) 430AD χ2-Test:d f=2 T=0.3(5%6.0)	Cabrera155:1795±18BP 610AD (68.2%) 670AD 560AD (95.4%) 700AD χ2 -Test:d f=1 T=2.9(5%3. 8)

Table 2: Calibrated radiocarbon dates from murex shells.
Tableau 2 : Dates radiocarbones calibrés des coquillages de murex

the murex shells and the human event of interest – purple dye production – (Van Strydonck *et al.*, 1999) than there is between this event and the bones or the charcoal found on site. Consequently the ideal radiocarbon sampling strategy should exhibit a good equilibrium between sample quality and sample integrity.

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