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A revised chronology of the lowest occupation layer of Pedra Furada Rock Shelter, Piauí, Brazil: the Pleistocene peopling of the Americas

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Abstract

The present work revisits the chronology of the archaeologically controversial Pedra Furada Rock Shelter of Southeast Piauí, Brazil, using an improved radiocarbon laboratory pre-treatment and measurements on charcoal samples. The procedure, known as ABOX-SC (acid–base–wet oxidation followed by stepped combustion), has previously been used to secure radiocarbon dates of >40 ka for the antiquity of human occupation of Australia and South Africa, and now has been applied to charcoal from the previously dated oldest occupation layer of the Pedra Furada site. Previous radiocarbon dating had obtained only lower limits of 40–45 ka BP for the Pedra Furada basal layer. Nine charcoal samples from well-structured hearths were subjected to the ABOX-SC procedure and their radiocarbon content determined by accelerator mass spectrometry. Measurements on five of the samples returned ages of greater than 56 ka BP, from graphites produced from ABOX pre-treated charcoal combusted at 910°C. Two other samples were greater than 50 ka BP. The remaining two samples were essentially completely combusted at 650°C, with no material surviving to make a 910°C CO_2 fraction. Their ages were 41.3 and 47.2 ka BP. Ages obtained from graphites generated from the 650°C combusted fraction are considered minimum ages.

1. Introduction

The Toca do Boqueirão do Sítio da Pedra Furada (BPF) site, at 8°51′ South and 42°33′200″ West, is a sandstone shelter located in the Maranhão–Piauí basin, 30 km NE from the town of São Raimundo Nonato, in Piauí State, Brazil (Fig. 1). It is one of 200 known painted rock-shelters located in a remote region inside of the Capivara National Park. The first excavations performed from 1978 to 1980, by the French–Brazilian archaeological mission in Piauí, had the objective of establishing a chronology for rock paintings in the region. On the basis of these paintings, the site was believed to be less then 10,000 years old. The discovery of well-structured hearths associated with abundant lithic industry (tools) in levels dated at 25 ka BP, however, made the Pedra Furada Rock Shelter one of the most controversial archaeological sites in South America, and launched a comprehensive exploration of the site (Guidon and Delibrias, 1985, 1986). The excavations furnished several thousand fragments of charcoal from hearths that, by their proximity to stone artifacts, were subsequently supposed to be of human origin. This assumption was based on both visual examination by experts on study of stone-tool typology (Guidon and Delibrias, 1985, 1986; Guidon and Arnaud, 1991; Guidon et al., 1996; Parenti, 1996) and thermoluminescence (TL) measurements on the hearth-stones (Parenti et al., 1990). The TL results demonstrated that the hearthstones were heated independently from the stones found outside the hearths in the same layer; thus, refuting the possibility that the stones were heated by natural fires (Bellomo, 1993). Until now, there has been no plausible explanation other than heating from human activity offered to reconcile the TL data

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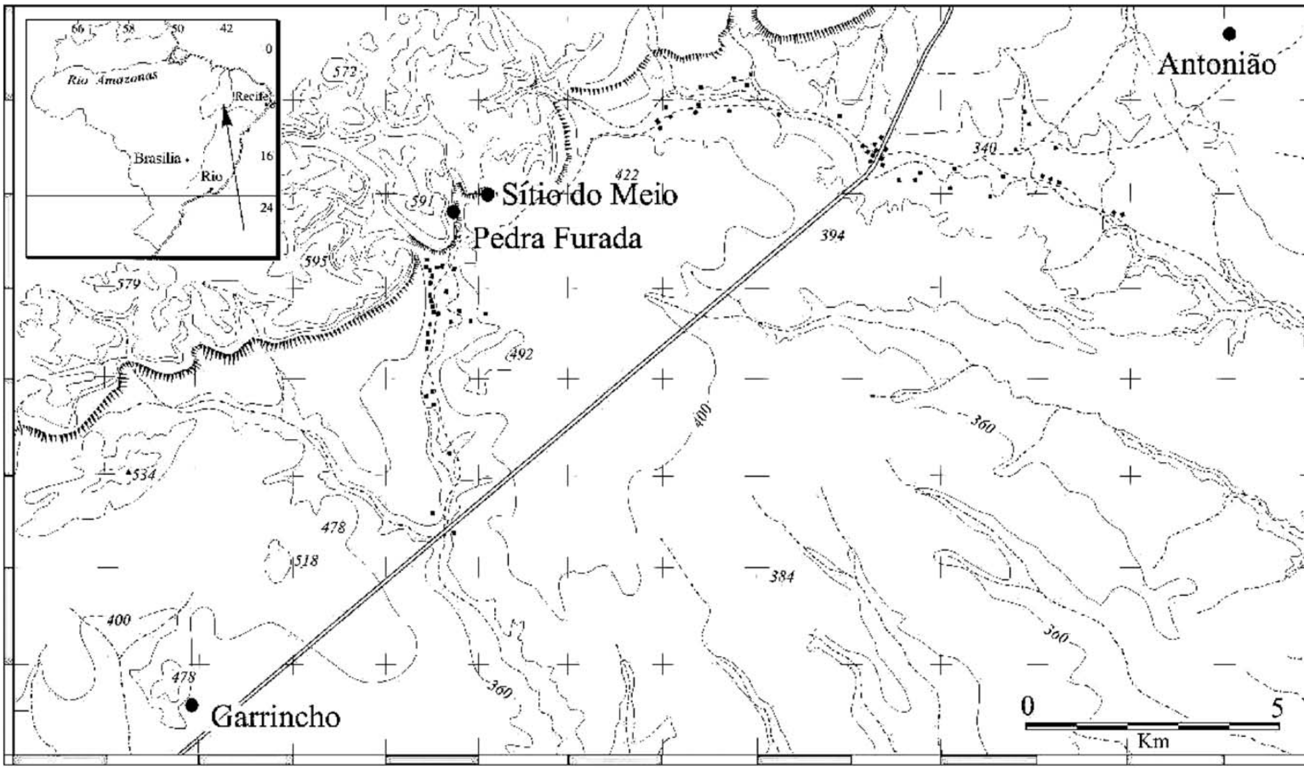

Fig. 1. Main sites of the São Raimundo Nonato region, Southern Piauí, where recent findings of Paleo-Indian skeletal evidence was found. The Garricho site, in which three fossilized human remains have been dated by Beta Analytic and gave an average result of 12,170 ± 40 BP years (BETA 136204) (Peyre, 1993; Peyre et al., 1998; Guidon et al., 2000, 2002). The Sitio do Meio, in which lithic industries and fireplaces have been recovered in the lowest unit, dated by radiocarbon at more than 18 ka BP and, the Antonião limestone rock-shelter, where pleistocenic fauna is associated with artifacts (Parenti et al., 2000).

(Parenti et al., 1990). Recently, another TL study on burnt quartz pebbles found in Pedra Furada site provided no further insight as to the origin of the archaeological remains. The TL ages estimated from the pebbles covered an inexplicable wide range (Valladas et al., 2003). Presently, the best evidence of human occupation for the basal layer is still the visual evaluation of stone-tool typology.

A comprehensive chronology at the Boqueirão da Pedra Furada site has been established with 46 reliable radiocarbon dates on charcoal excavated from different levels (Delibrias et al., 1988; Parenti, 1996, 2001). Two principal phases were identified: (a) Holocene and (b) Upper Pleistocene, which has been labeled the Pedra Furada phase. The Pedra Furada phase was divided into three layers (PF 1, 2 and 3) corresponding to age ranges >48–35, 32.2–25 and 21.4–14.3 ka BP. The Pedra Furada phase is represented by 32 dated charcoal samples. The resulting chronostratigraphy (Fig. 2) shows a sequence with no inversion of radiocarbon dates through the entire range displayed in the figure. The lack of date inversions shows that the profile was unperturbed. The consistent decrease in ages through the profile also demonstrates that contamination by older organic matter was unlikely (Bendnarik, 1989). Details of this chronology, stratigraphy and geological features have been published by Delibrias et al. (1988), Bednarik (1989), Guidon and Arnaud (1991) and Parenti (1996). A complete review on Pedra Furada stratigraphy and explanation on the anthropogenic origin of the archaeological remains was also published elsewhere (Parenti, 2001).

Such old dates for human habitation in South America were in profound disagreement with the established idea that the colonization of the Americas began between 12,000 and 14,000 years BP, when the existence of a land bridge and ice-free corridor allowed migrants from northeastern Asia (the Clovis people) to cross between Siberia and North America (Guthrie, 1984; Guidon and Arnaud, 1991). Despite the findings of late Pleistocene human occupations at several sites in the eastern tropical lowlands of South America, especially in central Brazil (Kipnis, 1998) and the Brazilian Amazon (Rooselvelt et al., 1996), this hypothesis has been accepted as doctrine for more than two decades, leading to a climate skeptical of revising the chronology of Pleistocene America. The remarkable finds of anthropogenic material at Monte Verde (MV) west of Puerto Montt, south-central Chile (Bednarik, 1989; Adovasio and Pedler, 1997; Dillehay, 1997; Meltzer, 1997; Nemecek, 2000), however, have called this conventional view into question. Two layers were

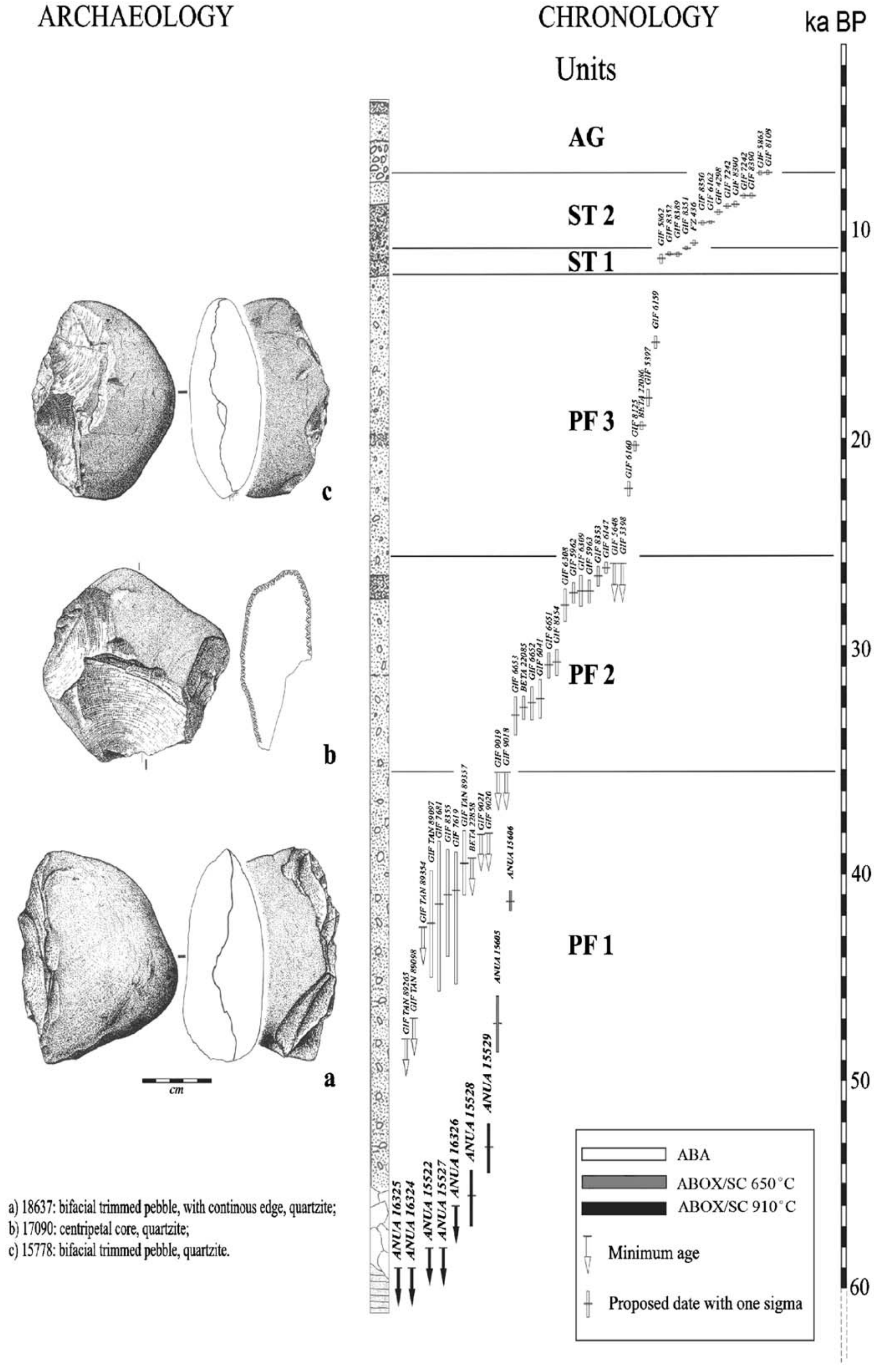

Fig. 2. Chronology and archaeological sequence of the Pedra Furada rock-shelter. The graphically represented artifacts (a–c) shown are examples of the lithic industries found in the respective layers associated with the charcoal pellets found in the well-structured hearths or in the immediate context of archaeological features. The lithic industry of the Pleistocene layers is obtained principally through monofacial techniques on quartz and quartzite materials, without any flaking patterns and minimal retouch. For a complete description on the study of the Boqueirão Pedra Furada industries the authors recommend the references (Parenti, 1996, 2001).

excavated at Monte Verde, MV1 and MV2 (MV2 is the layer dating to at least 12,500 years ago with undisputable findings, and MV1 is a deeper and older layer with two charcoal samples radiocarbon dated to around 30 ka BP; the older layer of this site is currently embroiled in a controversy regarding its validity) (Dillehay, 1997). Based on Monte Verde finds, a lively Clovis versus pre-Clovis debate and discussion of a possible coastal entry route for the origin of the first Americans have been stimulated (Fladmark, 1979; Bray, 1986; Guidon and Arnaud, 1991; Dixon, 1993, 2002; Gruhn, 1994; Bednarik, 1997; Meltzer and Dillehay, 1999; Guidon et al., 2002).

In 1993, the Pedra Furada site underwent strong criticism from three researchers after a visit by an interdisciplinary team of 13 members. The criticism was based on the claim that the artifacts found in Pedra Furada were from geofact origin (e.g. Meltzer et al., 1994). In 1996, this criticism was addressed in a paper published by the experts that excavated the site (Guidon et al., 1996). It seems that their reply was ignored by Meltzer and co-workers in subsequent publications (Meltzer, 1997; Meltzer and Dillehay, 1999). In a parenthetical response to speculations in the criticism, Dennell and Hurcombe (1995) reported their own unsuccessful attempt to recreate geofacts in way that was suggested by Meltzer et al. (1994). Dillehay (1999) addressed a comment on the validity of Pedra Furada in which the criticism of possible human occupation was limited to the deep layers (over 20 ka). In the present work, we refrain from elaborating on the discussion of Pedra Furada's authenticity as an early human occupation site, since our goal is to reframe the time reference of the basal layer. However, the claim that Pedra Furada is the "oldest human record" in South America has been stated in previously published material (Guidon and Delibrias, 1985, 1986; Bray, 1986; Delibrias et al., 1988; Bednarik, 1989; Parenti et al., 1990, 1996; Bahn, 1991, 1993; Guidon and Arnaud, 1991; Dennell and Hurcombe, 1995; Guidon et al., 1996, 2000; Parenti, 2001) and will not be discussed further in this paper.

Recent findings of Paleo-Indian skeletal evidence around Pedra Furada (Fig. 1), however, have been providing important clues about pre-Holocene peopling in the same region. They are: (a) the Garricho site, in which three fossilized human remains have been dated by Beta Analytic and gave an average result of 12,170±40 years BP (BETA 136204) (Peyre, 1993; Peyre et al., 1998; Guidon et al., 2000, 2002), (b) Sitio do Meio, in which lithic industries and fireplaces have been recovered in the lowest unit, dated by radiocarbon at more than 18 ka BP, and (c) the Antonião limestone rock-shelter, where Pleistocene fauna are associated with artifacts (Parenti et al., 2000).

While the human origin of the artifacts from the Pedra Furada site is still questioned in the deeper layers, they may eventually be significant. Consequently, it is important to put this site in the proper time frame. The goal of the present work was to revisit the basal layer of the Boqueirão da Pedra Furada phase (PF1). Four samples measured as part of the original dating from this oldest layer had yielded lower limits of 40–45 ka BP (Fig. 2). The previous results corresponded to the backgrounds achieved with conventional acid-wash or acid–base–acid (ABA) pre-treatments (Guidon and Arnaud, 1991).

In this work, charcoal samples were submitted to a more rigorous acid–base–wet oxidation (ABOX) pre-treatment, which was followed by a stepped combustion (SC) procedure to remove any residual contamination. This technique reliably removes contamination from charcoal and wood (Bird et al., 1999; Santos et al., 2001), enabling credible radiocarbon dating to ~55 ka BP, and has been employed to obtain secure radiocarbon dates beyond 40 ka for human sites in Australia (Fifield et al., 2001; Turney et al., 2001a, b) and Africa (Bird et al., 2003).

2. Sample selection

The nine charcoal samples investigated in this study (listed in Table 1, described below) were chosen to provide a reliable assessment of the lowest layer (PF1) in the Pedra Furada Rock Shelter and to determine whether coherent archaeochronological information associated with a stratigraphic sequence could be obtained on samples older than 40 ka BP.

Some of the samples chosen (sample code 17370, 18385(2), 18606) came from the immediate context of specific archaeological features. These samples are related with the hearths 49, 59, and the stone structure 100 (Parenti, 2001). The hearths were identified based on the presence of combustion remains, such as charcoal, ash, reddish and/or thermal fractured pebbles. The other charcoal samples were sieved from one specific square area (sample code 18275, 18276, 18278, 18390, 18385(1)). In this manner, we obtained an average dating of the whole sedimentary content of a single excavated unit, corresponding to 7–10 cm depth of filling of the 2 × 2 square meters (Table 1).

The majority of samples were closely associated with archaeological structures placed between the shelter wall and the drip line (Parenti, 2001). Some of these structures had been previously tested by TL analysis (Table 1). According to the excavators, the TL results indicated charcoal production in situ by human activities (Parenti et al., 1990).

3. Laboratory chemical sample preparation

Samples were stored in plastic bags and were later handled individually in a clean lab. All pre-treatment

Table 1
A list of the nine samples selected and studied in this work and their locations within the Pedra Furada site

Sample code #	Sector	Square	Unit	Closest to structure	Remarks
18276	East	Sieve C2	12 (1)	Hearth 61 (same)	
18275	East	Sieve C1	12 (1)	Hearth 61 (2 m)	
18390	East	Sieve D4	13 (1)	Hearth 57 (1 m)	Concentrated charcoals upon the hearth
17370	Trench 6	C6–D6	9 (6)	Hearth 49 (same)	Concentrated charcoals upon the hearth
18385 (2)	East	D2	13 (1)	Hearth 59 (same)	One (TL tested) heated pebble in the hearth[a]
18385 (1)	East	Sieve D2	13 (1)	Hearth 59 (same)	One (TL tested) heated pebble in the hearth[a]
18278	East	Sieve C4	12 (1)	Structure 99 (1 m)	Two heated pebbles in the structure
18606	Central	E2	14 (3)	Hearth 100 (same)	One (TL tested) heated pebble in the hearth[a]
17890	East-South		39		

[a] The heated pebble in the hearth was tested by TL analysis (Parenti et al., 1990).

steps were carried out in a laminar flow cabinet or fume hood. The vessels and implements that were in contact with the samples were rigorously pre-cleaned with acid-dichromate oxidant solution (0.1 M $K_2Cr_2O_7$ in 2 M H_2SO_4). The combustion and graphitization tubes were additionally baked at high temperature (pyrex −550°C and quartz and vycor tubes −950°C). The surfaces of the charcoal pellets were removed mechanically before the pellets were weighed and placed in 50 ml capped plastic Falcon® centrifuge tubes. The amount of charcoal used in the procedure was approximately 50 mg. In order to minimize the possibility of contamination from modern carbon due to handling of the samples, the charcoal pellets were not ground.

The ABOX pre-treatment involves the following steps:

(1) The tubes were filled with 20 ml of fresh 6 M HCl solution. An ultrasonic bath aided the pulverization of the pellets and the penetration of the solution into the charcoal fragments. After treatment for at least 1 h at room temperature, the samples were centrifuged and rinsed twice with Milli-Q® water.
(2) The tubes were filled with fresh 6 M HF solution and left to stand overnight (approximately 10 h), after which time the solution was rinsed as above. These two steps were carried out to remove acid soluble organic material and mineral impurities, respectively.
(3) Fresh 1 M NaOH (50 ml) were then added to each tube to remove the base soluble organic material. Samples were left standing in this solution overnight at room temperature before rinsing twice with Milli-Q® water.
(4) The wet oxidation step consisted of adding 30 ml of fresh acid-dichromate oxidant solution (0.1 M $K_2Cr_2O_7$ in 2 M H_2SO_4) to the tube in order to digest any remaining organic carbon on the samples. The tubes were capped and heated to 60.5°C in a temperature-regulated hot box. The acid-dichromate oxidant solution acts very aggressively. Most of the samples were subjected to this treatment for 5–6 h with a loss of 50% or more of the original material. For two of the samples (sample code: 18275 and 18276) (Table 1), however, it was necessary to reduce the time to 30 min and even then 80% of the sample was removed. Again, the supernatant was discarded, and the remaining fragments of clean charcoal were washed twice with warm Milli-Q® water, covered and allowed to dry in a warm oven (80°C).

The subsequent combustion and graphitization procedures were carried out on a vacuum line completely isolated from atmosphere by a second backing vacuum line and valves. Details may be found in Bird et al. (1999). The ABOX pre-treated samples were taken through a sequence of three combustion steps, at 330°C, 650°C and 910°C, with respective combustion times of 2, 1 and 12 h. The combustion time of the last step (910°C) was increased from the original 1 h (Bird et al., 1999) to at least 12 h in an attempt to ensure the complete reaction of other contaminant gases with the silver wire in the combustion tube, as it was suspected that the iron powder catalyst was being poisoned by some unremoved product at the shorter combustion time (Santos et al., 2001). Graphite targets were made from CO_2 produced at each of the two higher temperature combustion steps (i.e., 650°C and 910°C), while the CO_2 evolved at 330°C was discarded. This procedure allows the progressive removal of any contamination and its efficacy can be monitored from the targets produced. This allows a high degree of confidence on the final age assessment.

4. Radiocarbon AMS results and discussions

The graphite-plus-iron powder targets were pressed into aluminium sample holders and ratios of $^{14}C/^{13}C$ were measured by AMS using the 14UD accelerator at

Table 2
AMS radiocarbon dating of charcoal samples obtained from the lowest layer in the Pedra Furada Rock Shelter (PF1) using the ABOX-SC procedure

Laboratory number ANUA-[a]	Sample code	Fraction (°C)	Uncorrected pMC	Uncorrected pMC error	Uncorrected ^{14}C age (yr BP)[b]	Error (yr BP)	Blank corrected ^{14}C age[c,d] (yr BP)	Error (yr BP)
15606	18276	650	0.58	0.04	41,295	(+)540/(−)510		
15605	18275	650	0.28	0.05	47,200	(+)1490/(−)1255		
15529	18390	910	0.24	0.04	48,350	(+)1330/(−)1140	53,120	(+)3965/(−)2640
15604		650	0.28	0.03	47,175	(+)850/(−)770		
15528	17370	910	0.21	0.03	49,610	(+)1435/(−)1220	55,575	(+)5685/(−)3300
15603		650	0.21	0.03	49,480	(+)1160/(−)1020		
16326	18385 (2)	910	0.13	0.02	53,235	(+)1400/(−)1190	>56,000[e]	
15527	18385 (1)	910	0.11	0.02	54,825	(+)1189/(−)1035	>58,000[e]	
15602		650	0.17	0.04	51,174	(+)2090/(−)1220		
15522	18278	910	0.11	0.02	54,380	(+)1112/(−)980	>58,000[e]	
16324	18606	910	0.09	0.02	56,560	(+)1880/(−)1520	>59,000[e]	
16325	17890	910	0.10	0.02	55,570	(+)1590/(−)1320	>59,000[e]	

[a] Laboratory number ANUA- corresponds to the AMS code number of the measurements.
[b] Conventional radiocarbon ages without blank correction. δ^{13}C values were not measured. They have been assumed to be −25‰.
[c] A blank correction of 0.10 ± 0.02 pMC has been subtracted from the measured pMC values.
[d] Blank corrected values are not given for the 650°C fractions because the blank for these samples has not been defined, and could in principle be different from the 910°C blank.
[e] Limits are quoted at two standard deviations.

the Australian National University (laboratory code ANUA-). Complete results are shown in Table 2. The total procedural blank was determined to be 0.10 ± 0.02 pMC (equivalent to 55 ka BP) from multiple measurements on Ceylon graphite and charcoal achieved by the complete ABOX-SC procedure (Bird et al., 1999). Measurements on untreated Ceylon graphite yield apparent ages in excess of 60 ka.

Of the seven samples which yielded a 910°C fraction, five were indistinguishable from this procedural background, while the remaining two were within three standard deviations of the background. After subtraction of the blank correction, all seven correspond to ages greater than 50 ka. These are consistent with, but considerably older than, the earlier limits obtained for this level using more conventional pre-treatments and bulk combustion (Fig. 2). Three of the seven samples produced sufficient CO_2 at 650°C for a measurement, and in each case the 650°C fraction is slightly younger than, although statistically indistinguishable from, the 910°C fraction. This concordance between the two fractions gives confidence that between the pre-treatment procedure and discarding the 330°C combustion step, most if not all of the more modern contamination is removed.

As noted above, 80% of samples ANUA-15605 and 15606 (samples code: 18276 and 18275, respectively) (Table 2) was removed in 30 min by the oxidation step of the pre-treatment procedure. This suggests that the charcoal of these two samples was considerably more degraded than the other samples which survived for 5–6 h in the oxidation step with less loss of material. Further support for this view comes from the observation that both samples were essentially completely combusted at 650°C, with no material surviving to make a 910°C fraction. Measurement of the 650°C fraction yields an age for sample 18275 that is significantly younger than those obtained for the other seven samples, which is perhaps not surprising in view of the above. Sample 18276, on the other hand, yields an age which is close to those obtained for the other seven samples.

The sample coded 18385 was divided in two aliquots during the excavation process (Table 1) and sent to ANU in independent shipments. The results of uncorrected ^{14}C-age obtained on independent measurements of this sample (ANUA-15527 and 16326, 910°C fraction) (Table 2) are statistically indistinguishable, demonstrating the reproducibility of the method. The chronostratigraphy results from the samples of the Boqueirão da Pedra Fura phase 1 layer studied in this work are also in sequential concordance (Fig. 2).

5. Conclusions

From the total set of seven charcoal samples found in the hearth structures of the basal layer of Pedra Furada which survived the complete ABOX-SC procedure, five of the them proved to be beyond the limit of the technique itself, returning ages greater than 56 ka BP years. Finite ages of 53 and 55 ka years were obtained for the remaining two.

These results show that an improvement of at least 8 ka compares to previous data (Delibrias et al., 1988; Parenti, 1996, 2001). The ABOX-SC procedure demonstrated its ability to remove almost completely traces of extraneous contamination, which may be present as a result of exposure of the charcoal to the environment.

Regardless that some of the samples studied in this work return ages up to the ABOX-SC limit of the technique itself, the shift in age achieved for these samples contaminated in situ still impressive when one is near the radiocarbon age limit. Two of the new values have helped to obtain limit ages for the basal Layer in the Pedra Furada Rock Shelter and, so far they are the oldest radiocarbon results obtained for any South America archaeological site.

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