CROSS-DATING (Th/U-^{14}C) OF CALCITE COVERING PREHISTORIC PAINTINGS AT SERRA DA CAPIVARA NATIONAL PARK, PIAUI, BRAZIL

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ABSTRACT. The question of when the first humans arrived in the New World has been a bone of contention for several decades. Similarly, the age of rock paintings has been heatedly debated. Settlements in the Serra da Capivara National Park have been dated to between 5 kyr and >50 kyr, which is far older than the Clovis barrier. Moreover, calcite formation on a rock-wall painting in a rockshelter yielded thermoluminescence (TL) and electron paramagnetic resonance (EPR) ages older than 35 kyr BP (Watanabe et al. 2003). In an attempt to contribute to this ongoing debate, we have studied calcite deposits covering prehistoric paintings from several rockshelters (Toca da Bastiana, Toca do Serrote de Moendas, and Toca da Gameleirinha [Pedra pintada]). Coupled AMS (accelerator mass spectrometry) ^{14}C and MC-ICPMS (multi-collector inductively coupled plasma mass spectrometry) ^{230}Th/U dating was performed in Toca da Gameleirinha. The ages obtained for these calcites are younger than 12 kyr and suggest that the paintings could be more recent than proposed by previous studies.

INTRODUCTION

In South America, as in the Old World, cave paintings and engravings are among some of the earliest examples of art and human symbolic behavior. However, the ages of these practices remain highly uncertain especially as no stylistic convergence can be established between the different sites scattered over a vast territory. In addition, the date of the arrival humans on the American continent, which underlies all assumptions for the age of the paintings, remains a controversial subject (Lavallée 2000; Rothhammer and Dillehay 2009; Holliday 2009). In almost all the American sites, the paintings lack organic pigments or binders suitable for accelerator mass spectrometry (AMS) radiocarbon dating. In the absence of any direct dating, archaeologists have reasonably assigned the age of the paintings to the age of dated human settlements in the excavations near the rock walls.

The Serra da Capivara National Park (SCNP) (between 8°26′50″ and 8°54′23″S and 42°19′47″ and 42°45′51″W) is known worldwide for its numerous ancient rock-wall paintings. The park is situated in northeastern Brazil near the town of São Raimondo Nonato, 220 km south of Floriano and 523 km from Teresina. The main body of the park is the Serra do Congo massif and the central Chapada da Capivara in the state of Piauì. The SCNP is located on a Silurian and Devonian sandstone formation (Parenti 2001), with small limestone formations (*morros* or *serrotes*) of Precambrian age emerging as island mountains above the sandstone plateau (*chapada*) and the pediment. These very fine gray metamorphosed limestones are deeply eroded, with many cavities, rockshelters, and caves. Over 300 archaeological sites have been found within the park, the majority including rock and wall paintings.

Systematic excavations by Niède Guidon and Fabio Parenti at archaeological sites provided more than 55 ^{14}C ages of charcoals collected from several layers at the rockshelters ranging from 6200 to more than 52,000 ^{14}C yr BP (Guidon and Délibrias 1986; Guidon and Arnaud 1991; Parenti et al.

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1996; Parenti 2001; Santos et al. 2003). Watanabe et al. (2003) at Toca da Bastiana (SCNP) and Sastry et al. (2004) at Montalvania in Minas Gereis dated calcite covering wall paintings and calcite covering carvings by thermoluminescence (TL) and electron paramagnetic resonance (EPR) techniques, respectively. Both TL and EPR dating of this calcite gave an age ranging between 48 and 55 ± 5 kyr BP for the Montalvania site (otherwise undated), and TL gave an age between 35 to 43 ± 5 ka BP for Toca da Bastiana, in agreement with the ages of the oldest settlements in the SCNP.

Conversely, Rowe and Steelman (2003) reported a ^{14}C measurement of calcium oxalate extracted from the calcite layer dated by Watanabe et al (2003), which "yielded only 2,490 ± 30 ^{14}C yr BP for a minimum age." Using plasma-chemical extraction and accelerator mass spectrometry (AMS) ^{14}C dating a "direct" date for the red "paintings" associated with the calcite layer yielded 3730 ± 90 ^{14}C yr BP. In addition, they reported 4 dates on paintings from Bastiana as well as 4 more from Toca do Sitio do Meio, Toca do Extreme, and Pedra Furada, all of which fell between about 1200 to 3600 ^{14}C yr BP. These new results are problematic insofar as they are younger than the most recent occupations dated in the SCNP. If the very old dates are correct, it implies that the paintings in the SCNP are the oldest artistic manifestations of humanity. On the other hand, the recently obtained youngest ages suggest the presence of a cultural phase that has left no trace in the excavations. Such a fundamentally contradictory hypothesis needs to be tested.

MATERIAL AND METHODS

We used ^{14}C dating of thin calcite overgrowths covering paintings in Toca da Bastiana. Similar overgrowths formed on the surfaces of paintings of the Toca da Gameleirinha in the SCNP were cross-dated using both ^{14}C and uranium-series disequilibrium, as suggested by Plagnes et al. (2003). Three rockshelters (Toca da Bastiana, Toca do Serrote de Moendas, and Toca da Gameleirinha [Pedra pintada]) were chosen in these limestone formations. At Toca da Bastiana, the calcite deposits are thin and were sampled directly on paintings and near the sampling carried out by Watanabe several years earlier. We sampled the most marginal parts of the paleo-artwork in order to avoid any prejudice to the artistic quality of the painted design.

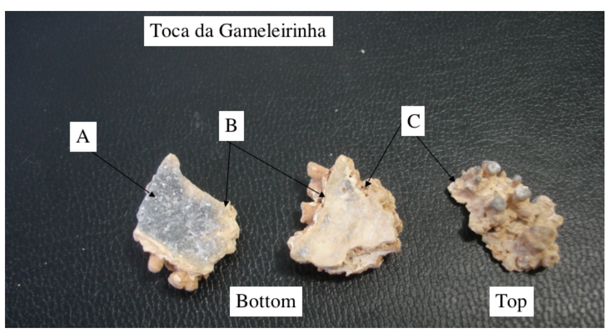

Figure 1 Photo of the first calcite sample that was thick enough to be divided into 3 subsamples: Ech A calcite at the bedrock contact; Ech B compact calcite fraction overlying contact with the bedrock; and Ech C outer calcite with cauliflower-like surface.

At Toca do Serrote de Moendas, a calcite sampling superimposed onto a black painting was first performed in 2006, and the calcite directly in contact with the bedrock was analyzed. A second sampling of the compact calcite fraction, overlying the contact with bedrock, was performed in 2008. At Toca da Gameleirinha, 2 thick calcite deposits overlying paintings were selected. The first one was thick enough for 3 subsamples to be selected for analysis: Ech A calcite at the bedrock contact; Ech B compact calcite fraction overlying contact with the bedrock; and Ech C outer calcite with a cauliflower-like surface (see Figure 1). For the second sample, only 2 levels were analyzed, Ech D and Ech E, which are similar to Ech B and C, respectively.

^{14}C Dating

Calcite samples were finely crushed, then washed in distilled water using an ultrasonic bath. The grains were then lightly etched using 0.01N HNO_3 and then rinsed with distilled water. Under vacuum, the calcite was reacted with phosphoric acid and the evolved CO_2 was reduced to obtain graphite targets prepared following the method described by Tisnérat-Laborde et al. (2001). Analyses were performed using the Saclay AMS facilities (Artemis) and results are expressed as conventional ages following Stuiver and Polach (1977). Calibrated ages are expressed as cal BP with a confidence level of 95.4% (2σ). These ages need to be corrected for the dead carbon fraction (dcf) coming from the dissolution of the carbonate host rock. The dcf is generally considered as stable, ranging from 5 to 20% of total carbon for contemporaneous carbonate deposits at the same site (Genty and Massault 1997; Genty et al. 1999, 2001; Beck et al. 2001; Plagnes et al. 2003).

^{230}Th/U Dating

The chemical preparation of the samples was similar to the method described by Douville et al. (2010). Following mechanical cutting and cleaning of visible non-carbonate particles, the calcite fragments were rinsed in Milli-Q™ water and dilute HNO_3 (0.1N) for 10 min in an ultrasonic bath, then dried overnight in an oven at 60 °C. The calcite samples were then weighed and dissolved in 6N HNO_3 acid in a clean Teflon® vial containing a known quantity of a ^{229}Th, ^{233}U, and ^{236}U triple spike. The dissolved sample-spike mixture was dried on a hot plate overnight and redissolved in 3N HNO_3. Thorium and uranium were extracted and purified from the sample solutions using the ion exchange resin U-TEVA according to Douville et al. (2010). Finally, the Th and U fractions were reduced to <10 µL solution on a hot plate and redissolved in a mixture of 0.1N HNO_3 and 0.01 HF.

To perform simultaneous measurements of all Th and U isotopes, the Th fractions were doped with a small amount of the sample U fractions. Isotopic measurements of ^{229}Th, ^{230}Th, ^{232}Th, ^{233}U, ^{234}U, ^{235}U, ^{236}U, and ^{238}U were subsequently carried out on a Neptune Plus MC-ICPMS instrument located at Laboratoire des Sciences du Climat et de l'Environnement using a standard sample bracketing technique with samples interspaced with appropriate instrumental and chemical blank solutions. ^{234}U and ^{230}Th were measured on the central electron multiplier (IC1) using a peak-jumping routine. All other isotopes (^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{229}Th, and ^{232}Th) were simultaneously collected on the Faraday cups. Sample standard measurements were interspaced with measurements of the ion counter to Faraday cup yield as well as hydrate interferences and peak tailing to correct for machine abundance sensitivity. Data reprocessing was performed to remove instrumental blanks and to correct for mass fractionation using the natural ^{235}U/^{238}U atomic ratio (137.88). Hydrate and peak tailing interferences were removed based on the mass ratio 239/238 and half-mass measurements. Finally, isotopic ratios were normalized to the isotopic compositions of the secular equilibrium standard (Harwell Uraninite) HU-1 (Cheng et al. 2000) used for bracketing. This technique provides accurate and reproducible measurements of ^{230}Th/^{238}U, ^{234}U/^{238}U, ^{230}Th/^{232}Th, ^{235}U/^{236}U, and ^{229}Th/^{230}Th at <2‰ precision. Full procedural chemical blanks were ultimately considered for final data adjustment, and age calculation from the isotopic data was based on iterative age estimation (Ludwig and Titterington 1994) propagating all analytical errors into the Monte Carlo age simulation. Finally, ages need to be corrected for the (^{230}Th/^{232}Th) ratio from non-carbonate contamination and possible residual contamination from excess ^{230}Th in the bedrock (see below).

RESULTS AND DISCUSSION

^{14}C dates are reported in Table 1 and the Th/U series in Table 2. ^{14}C ages were calibrated using the CALIB v 6.0 program applying the Southern Hemisphere correction except for 2 samples older than 10,000 ^{14}C yr BP (Stuiver and Reimer 1993; McCormac et al. 2004). Ages ranged between 1770 and 4390 ± 30 ^{14}C yr BP (1554–5031 cal BP), and between 4520 ± 30 and 11,805 ± 35 ^{14}C yr BP for Toca da Bastiana and Toca da Gameleirinha, respectively. At Toca do Serrote de Moendas 2 contrasting dates were obtained: 31,860 ± 210 and 1590 ± 30 ^{14}C yr BP. The older of these 2 dates, ~11.8 yr BP, and the ^{14}C age from Toca da Gameleirinha were both obtained for calcite samples in contact with the bedrock, where the calcite is deposited on the more or less eroded porous surface of the bedrock. The evidence supports the idea that the calcite collected at these contacts may be a mixture of old limestone and recent deposits, and may have yielded anomalously old ages. This hypothesis is supported by the quite different uranium contents and Th/U age estimation of sample Ech A, which suggest that our sample includes in fact a non-negligible amount of carbonate from the bedrock. The higher δ^{13}C values of Ech A also point to such a contamination. These 2 results are therefore not included in the discussion.

The dcf in the sites studied here is unknown, but taking into account the youngest dates (1500 ^{14}C yr BP) and the open-air location of the rockshelters, the dcf is certainly low and could range between 0 and 10%, giving an age correction ranging between 0 and 850 yr. For the present discussion, we selected dcf values of 0 and 5% (400 yr), which seem to be the most probable estimated values.

Whatever the dcf corrections are, the minimum median ages obtained are recent, spanning from 1425 to 4906 (dcf = 0) or 1028 to 4499 (dcf = 5%) yr BP. These results agree with those obtained by Rowe and Steelman (2003), who obtained 2430 ± 30 ^{14}C yr BP as the minimum ^{14}C age for calcium oxalate extracted from calcite dated by Watanabe et al. (2003) and 3790 ± 90 ^{14}C yr BP for direct ^{14}C dating using plasma chemical extraction and AMS ^{14}C dating. Results concerning other paintings at Toca da Batiana range between 1880 ± 60 and 3320 ± 50 ^{14}C yr BP. Nearby shelters gave similar results: Toca do Sitio do Meio (2700 ± 110 ^{14}C yr BP); Pedra Furada (2120 ± 110 and 3570 ± 50 ^{14}C yr BP); and Toca do Extreme (1230 ± 50 ^{14}C yr BP).

Toca da Gameleirinha provides older ^{14}C ages, ranging between 9275 ± 30 and 4520 ± 30 ^{14}C yr BP (median dates 10,376 and 5135, or 9863 and 4552 cal BP for dcf = 0 and dcf = 5%, respectively). The compact calcite fractions overlying contact with the bedrock (Ech B and Ech D) have similar ages, slightly less than 10,000 ^{14}C yr. The external calcites with a cauliflower-like surface in levels Ech C and EchE differ more markedly, with ages of 7165 ± 30 and 4520 ± 30 ^{14}C yr BP, respectively. Nevertheless, taken at face value, these ages suggest that calcite deposits occurred during the first half of the Holocene.

To test the consistency and representativeness of these dates, we proceeded, following Plagnes et al. (2003), to carry out Th/U datings of the thicker calcite sample. It is important to note, however, that in contrast to ^{14}C dating, U-series dating of such small and often poorly developed calcite deposits may suffer from contamination by unsupported ^{230}Th or ^{234}U derived from sources other than decay of ^{238}U in the sample, such as from meteoric water or detrital mineral or organic inclusions. Alternatively, several calcite generations of different ages are possible without leaving any visible trace. If such processes occurred, the measured disequilibrium dates derived could be either younger or older than the mean actual age of deposition of the encrusting calcite.

Table 1 Radiocarbon dates of calcite deposits overlying paintings. Dates given at the 95.4% confidence level.

Lab code SacA-	Sample reference	$\delta^{13}C$ (‰)	Conv. age BP dcf = 0%	Calibrated dates BP and (median) dcf = 0%	dcf = 5%
Toca da Bastiana 1					
7480	Calcite overlying external paintings 1	–9.6	2455 ± 30	2342–2695 (2430)	1872–2041 (1949)
7482	Calcite overlying external paintings 3	2.7	1770 ± 30	1544–1704 (1622)	1178–1296 (1252)
7483	Calcite overlying external paintings 4	–3.2	4390 ± 30	4839–5031 (4906)	4418–4783 (4499)
Toca da Gameleirinha (Pedra pintada)					
11845	Ech A Calcite at the bedrock contact	–0.60	11,805 ± 35	13,471–13,789	
11846	Ech B Compact calcite fraction overlying contact with bedrock	–3.80	9275 ± 30	10,258–10,498 (10,376)	9696–10,149 (9863)
11847	Ech C Outer calcite with cauliflower surface	–1.80	7165 ± 30	7852–8007 (7943)	7507–7656 (7579)
11848	Ech D Compact calcite fraction overlying contact with bedrock	–1.30	8870 ± 30	9690–10,147 (9851)	9312–9524 (9453)
11849	Ech E Outer calcite with cauliflower surface	3.10	4520 ± 30	5050–5305 (5135)	4430–4801 (4552)
Toca do Serrote de Moendas					
11850	Calcite overlying external paintings 5	–6.10	1590 ± 30	1352–1519 (1425)	964–1168 (1028)
7484	Compact calcite fraction overlying contact with bedrock	–6.2	31,860 ± 210	35,520–36,822	

Table 2 Results of U-series disequilibrium dating for samples of Toca da Gameleirinha. Three sets of ages are given, uncorrected, as well as *Model 1, and **Model 2, which are corrected for detritus. *Model 1 uses a $^{230}Th/^{232}Th$ activity ratio of contamination of 2.3, which is the regression mean between the 2 calcite samples (isochron plot); **Model 2 uses a $^{230}Th/^{232}Th$ activity ratio of contamination of 5.5 ± 0.5 needed to obtain the reservoir uncorrected but calibrated ^{14}C age of the 2 samples.

Reference	$[^{238}U]$	$\delta^{234}Um$	$(^{230}Th/^{232}Th)$	$[^{232}Th]$	$^{230}Th/^{238}U$	Age (kyr)	$\delta^{234}U(0)$ (‰)	$\delta^{234}U(0)$ (‰)	Model 1 age (kyr)*	Model 2 age (kyr)**
Ech A	2.473 ± 0.002	243.4 ± 1.1	72.01 ± 0.09	62.101 ± 0.056	0.63396 ± 0.00075	75.78 ± 0.23	301.4 ± 1.3			
Ech B	10.813 ± 0.012	486.6 ± 0.6	26.23 ± 0.04	205.084 ± 0.216	0.17419 ± 0.00023	13.53 ± 0.03	504.9 ± 0.6	503.1 ± 0.7	12.28 ± 0.186	10.56 ± 0.289
Ech B	10.792 ± 0.013	487.3 ± 0.8	26.17 ± 0.07	205.460 ± 0.264	0.17445 ± 0.00045	13.53 ± 0.05	506.3 ± 0.8	504.5 ± 0.8	12.28 ± 0.206	10.56 ± 0.309
Ech C	4.423 ± 0.006	488.1 ± 0.7	7.68 ± 0.02	372.256 ± 0.720	0.22644 ± 0.00045	17.87 ± 0.05	513.4 ± 0.7	505.3 ± 1.3	12.24 ± 0.761	4.81 ± 1.163

Three calcite samples covering paintings from Toca da Gameleirinha were investigated using mass spectrometry U-series dating as described above. One sample is clearly in contact with the bedrock (Ech A), yielding a very high ^{14}C age. This sample must be considered as a mixture of carbonate bedrock and calcite overgrowth. The other 2 calcite precipitates of distinct shapes (Ech B compact calcite, Ech C cauliflower-like structure) were considered more likely to give useful ages. U concentrations of all 3 samples were high, ranging from 2.5–11.8 µg/g, which most likely indicates precipitation of high Mg calcite from meteoric water enriched in U. The lowest concentration was observed in the sample with a suspected admixture of bedrock carbonate. The U-isotopic composition, expressed in per mil deviation from radioactive equilibrium (^{234}U), ranged from 240 to 489‰ and is thus within the range of typical calcite deposited from meteoric groundwater containing excess ^{234}U. Typically, ^{232}Th is used to detect and correct for exogenous non-carbonate contamination in such deposits, which generally has low and variable $^{230}Th/^{232}Th$ ratios. Mineral detritus would be expected from mean continental crust values to carry a ($^{230}Th/^{232}Th$) ratio of 0.79. In our case, the sample containing a significant amount of bedrock had the highest measured ($^{230}Th/^{232}Th$) activity ratio, which may indicate that the bedrock carbonate is far from radioactive equilibrium as discussed below. Raw U-series ages, i.e. without correcting for exogenous ^{232}Th and ^{230}Th contamination from detritus or other sources, yielded values ranging from 76,000 to 13,500 yr (Table 2), which are in disagreement with the measured ^{14}C ages.

After classical correction using an assumed (Bulk Earth) detrital contamination, no significant change in the age data was observed. Using an alternate isochron approach for the samples Ech B and Ech C to measure and then correct for exogenous ^{230}Th, a $^{230}Th/^{232}Th$ ratio of 2.3 was obtained for the contaminant and both samples are presumed to have an identical age (presumption for an isochron) of 12,400 yr (Table 2). This age is closer to the ^{14}C ages for these samples, but a moderate discrepancy between ^{14}C and Th/U dating remains. Thus, the question arises whether the Th/U age is affected by other than detrital or small excess Th carrying contamination or whether ^{14}C ages are somehow erroneous. The likeliest assumption is that U and Th in such small deposits is disturbed since both are present in trace concentrations in calcite and easily affected by addition from the surrounding environment carrying both isotopes at higher concentrations and likely far from radioactive equilibrium. If bedrock carbonate is rich in U but has been significantly weathered, ^{238}U can be depleted while at the same time the ^{230}Th activity may be in excess because Th is generally less mobile than U. This would result in lower U isotopic compositions of a sample of calcite with admixed bedrock and a high $^{230}Th/^{238}U$ ratio as well as a higher $^{230}Th/^{232}Th$ ratio. Since the pure bedrock was not analyzed, quantitative estimates of the amount of bedrock cannot be made from isotopic measurements, but a simple mixing calculation would yield up to 80% bedrock ^{230}Th in sample Ech A. Furthermore, one may assume that even the other calcite deposits contain minor amounts of particles from the bedrock or have precipitated from meteoric waters in contact with the bedrock. In the first scenario, the samples would be contaminated with material having a high $^{230}Th/^{238}U$ ratio and a low $^{234}U/^{238}U$ ratio. In the latter case, meteoric water percolating towards the carbonate precipitation site may be subject to the uptake of ^{234}U(^{234}Th) and ^{230}Th from alpha recoil occurring in the bedrock. This process would again yield excess ^{230}Th but also excess ^{234}U. From the small data set, such processes can neither be distinguished nor correctly modeled. Therefore, we simply tested through an iterative approach at which contaminant ($^{230}Th/^{232}Th$) ratio both dating approaches (Th/U and ^{14}C) would yield identical ages for samples Ech B and Ech C within error margins, assuming the ^{14}C dating to be correct (Model 2 age). Very similar ages can be obtained through a correction with a ($^{230}Th/^{232}Th$) ratio of 5.5 for both calcite samples. Such a ratio is high but remains within a possible range.

Overall, Th/U dating clearly appears to support the findings regarding the erroneously high ^{14}C age of Ech A, and through a reasonable correction both dating techniques can be found to agree, pointing to early an Holocene to mid-Holocene deposition of the calcite overgrowth of the Toca da Gameleirinha rockshelter.

CONCLUSIONS

^{14}C ages obtained for these calcites do not constitute strictly a minimum age since no dead carbon corrections were performed and a small probability remains that the true age of painting fall in between the bulk (apparent) age and the dfc-corrected age. At Toca da Bastiana and Toca do Serrote de Moendas, the dead carbon is low, <1500 ^{14}C yr, suggesting that these paintings were made at least later than the upper Holocene, in good agreement with Rowe and Steelman (2003) and significantly rejuvenating minimum ages obtained by Watanabe et al. (2003). At Toca da Gameleirinha, Th/U estimates were slightly higher than ^{14}C ones but suggest that the paintings could be more recent than proposed by previous studies. Ages between 8–12 kyr are compatible with the early stage of the Serra Talhada phase and the final stage of the Pedra Furada phase. Nevertheless, the calcite overgrowth could have grown there long after the paleo-artwork was applied.

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REFERENCES

Beck JW, Richards DA, Edwards RL, Silverman BW, Smart PL, Donahue DJ, Hererra-Osterheld S, Burr GS, Calsoyas L, Jull AJT, Biddulph D. 2001. Extremely large variations of atmospheric ^{14}C concentration during the last glacial period. *Science* 292(5526):2453–8.

Cheng H, Edwards RL, Hoff J, Gallup CD, Richards DA, Asmerom Y. 2000. The half-lives of uranium-234 and thorium-230. *Chemical Geology* 169(1–2):17–33.

Douville E, Sallé E, Frank N, Eisele M, Pons-Branchu E, Ayrault E. 2010. Rapid and accurate U-Th dating of ancient carbonates using inductively coupled plasma-quadrupole mass spectrometry. *Chemical Geology* 272(1–2):1–11.

Genty D, Massault M. 1997. Bomb ^{14}C recorded in laminated speleothems: calculation of dead carbon proportion. *Radiocarbon* 39(1):33–48.

Genty D, Massault M, Gilmour M, Baker A, Verheyden S, Kepens E. 1999. Calculation of the past dead carbon proportion and variability by the comparison of AMS ^{14}C and TIMS U/Th ages on two Holocene stalagmites. *Radiocarbon* 41(3):251–70.

Genty D, Baker A, Massault M, Proctor C, Gilmour M, Pons-Branchu E, Hamelin B. 2001. Dead carbon in stalagmites: carbonate bedrock paleodissolution vs. ageing of soil organic matter. Implications for ^{13}C variations in speleothems. *Geochimica Cosmochimica Acta* 65(20):3443–57.

Guidon N, Arnaud B. 1991. The chronology of the New World: two faces of one reality. *World Archaeology* 23(2):167–8.

Guidon N, Délibrias G. 1986. Carbon-14 dates point to man in the Americas 32,000 years ago. *Nature* 321(6072):769–71.

Holliday VT. 2009. Geoarchaeology and the search for the first Americans. *Catena* 78(3):310–22.

Lavallée D. 2000. *The First South Americans*. Salt Lake City: University of Utah Press. 260 p.

Ludwig KR, Titterington DM. 1994. Calculations of $^{230}Th/U$ isochrons, ages and errors. *Geochimica et Cosmochimica Acta* 58(22):5031–42.

McCormac FG, Hogg AG, Blackwell PG, Buck CE, Higham TFG, Reimer PJ. 2004. SHCal04 Southern Hemisphere calibration 0–11.0 cal kyr BP. *Radiocarbon* 46(3):1087–92.

Parenti F. 2001. *Le gisement quaternaire de Pedra Furada (Piauí, Brésil). Stratigraphie, chronologie, évolution culturelle.* Paris: Editions Recherche sur les Civilisations. 445 p.

Parenti F, Fontugne M, Guerin C. 1996. Pedra Furada in Brazil and its presumed evidence: limitations and potential of the available data. *Antiquity* 70(268):416–21.

Plagnes V, Causse C, Fontugne M, Valladas H, Chazine J-M, Fage L-H. 2003. Cross dating (Th/U-^{14}C) of calcite covering prehistoric paintings in Borneo. *Quaternary Research* 60(2):172–9.

Rothhammer F, Dillehay TD. 2009. The Late Pleistocene colonization of South America: an interdisciplinary perspective. *Annals of Human Genetics* 73(5):540–9.

Rowe MW, Steelman KL. 2003. Comment on "Some evidence of a date of first humans to arrive in Brazil." *Journal of Archaeological Science* 30(10):1349–51.

Santos GM, Bird MI, Parenti F, Fifield LK, Guidon N, Hausladen PA. 2003. A revised chronology of the lowest occupation layer of Pedra Furada Rock Shelter, Piaui, Brazil: the Pleistocene peopling of the Americas. *Quaternary Science Reviews* 22(21–22): 2303–10.

Sastry MD, Sullasi HSL, Camargo F, Watanabe S, Prous APP, Silva MMC. 2004. Dating sediment deposits on Montalvanian carvings using EPR and TL methods. *Nuclear Instruments and Methods in Physics Research B* 213:751–5.

Stuiver M, Polach HA. 1977. Discussion: reporting of ^{14}C data. *Radiocarbon* 19(3):355–63.

Stuiver M, Reimer P. 1993. Extended ^{14}C data base and revised CALIB 3.0 ^{14}C age calibration program. *Radiocarbon* 35(1):215–30.

Tisnérat-Laborde N, Poupeau JJ, Tannau JF, Paterne M. 2001. Development of an automated system for routine preparation of carbonate samples. *Radiocarbon* 43(2A):299–304.

Watanabe S, Ayta WEF, Hamaguchi H, Guidon N, La Salvia ES, Maranca S, Baffa O. 2003. Some evidence of a date of first humans to arrive in Brazil. *Journal of Archaeological Science* 30(3):351–4.