

Radiocarbon Calibration by Means of Mass Spectrometric 230Th/234U and 14 C Ages of Corals: An Updated Database Including Samples from Barbados, Mururoa and Tahiti

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RADIOCARBON CALIBRATION BY MEANS OF MASS SPECTROMETRIC 230TH/234U AND 14C AGES OF CORALS: AN UPDATED DATABASE INCLUDING SAMPLES FROM BARBADOS, MURUROA AND TAHITI

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ABSTRACT. As first shown by Bard et al. (1990a), high-precision 230Th-234U ages can be used successfully to calibrate the radiocarbon time scale beyond the high-precision tree-ring calibration that now reaches 11,900 cal BP (Kromer and Spurk 1998). Using mass spectrometric techniques, we measured 14C and 230Th ages on new samples collected from boreholes drilled off the islands of Tahiti and Mururoa (French Polynesia) in order to complement the database previously obtained on Barbados corals (Bard et al. 1990a, 1993).

METHODS

New 230Th/234U ages for Tahiti and Mururoa samples (Table 1) were measured with a VG-54-30 thermal ionization mass spectrometer (TIMS) fitted with an ion-counting Daly detector, at CEREGE (Aix-en-Provence). The chemical separations were similar to those previously described (Bard et al. 1990b). The 2σ precision of the ²³⁰Th ages ranges from 30 to 60 yr, for ages between 8000 and 14,000 230Th yr BP.4 This represents an improvement by a factor of 2 to 3 over the performance obtained on Barbados corals by single collection and analog Daly detector on an MM30 mass spectrometer (Table 1; Bard *et al.* 1990a,b). The precision of the ages was checked by measuring numer-
ous replicates (Bard *et al.* 1996). In particular, we performed five analyses of different pieces of the same coral specimen (sample P7-7: 10,995 \pm 40, 11,005 \pm 30, 11,025 \pm 30, 10,995 \pm 30 and 10,995 \pm 30 ²³⁰Th yr BP; ages are rounded to the nearest 5 yr). The five ²³⁰Th/²³⁴U ages agree with each other within the 2σ uncertainties, with an overall 2σ uncertainty on the mean of 12 yr and a maximum difference between replicates of *ca*. 30 yr.

Following Ludwig et al. (1992) and Stirling et al. (1995), the ²²⁹Th $/233U$ ratio of our mixed spike was calibrated against the uraninite standard HU1 assumed to be at exact secular equilibrium. This calibration was shown to be accurate within 5% by means of gravimetric U and Th standards. This agreement is satisfactory if one takes into account the overall uncertainty on the half-lives of 234 U and 230Th (2%o and 8%o, respectively; see Ludwig et al. 1992).

Th samples are loaded with colloidal graphite on single-zone refined Re filaments and U samples on Re-Ta triple filaments. 234U/238U ratios are measured in dynamic multicollection mode: 233U, 234U and 235U ion beams are measured with the Daly ion counting detector, whereas 235U and 238U ion currents are measured with Faraday cups. Correction for isotopic fractionation is performed by nor malizing the measured $^{238}U/^{235}U$ atomic ratio to the natural value (137.88). Faraday/Daly gain is monitored with 235U signals during each measurement block in order to correct for possible shifts of the gain.

The external precision on individual values of $\delta^{234}U_i$ (= [initial²³⁴U/²³⁸U – 1] × 1000) is on the order of 2% (at 2σ), as shown by repeated measurements of standards. The accuracy has been checked by

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⁴A11 radiometric ages expressed here as "BP" are relative to a fixed present of 1950.

TABLE 1. Comparison of Ages Obtained on Corals by AMS 14C and TIMS 230Th

		U/Th age		${}^{14}C$ age			
	Sample code	(yrBP)	$\pm 2\sigma$ Th	(yrBP)	$\pm 2\sigma$ ¹⁴ C	$\Delta^{14}C$	$\pm 2\sigma \Delta$
	Barbados						
	RGF B-56*+	773	10	960	90	-26	11
	RGF 7-4-2	7460	80	6605	150	83	23
	RGF 7-5-5	8450	50	7640	160	74	22
	RGF 7-12-2*+	9265	60	8305	120	91	18
	RGF 7-16-2	9730	50	8750	170	92	24
	RGF 7-27-4	11090	70	9710	210	142	31
	RGF 12-5-2†	11590	60	9980	170	173	26
$\gamma_{\!\ell_{\mathcal{C}}}$	RGF 12-6-7†	11530	70	10095	160	148	25
	RGF 12-9-5†	12260	90	10220	130	235	24
	RGF 12-16-5*†	13100	80	11270	100	199	19
	RGF 12-21-6	13700	170	11710	200	221	39
	RGF 12-21-10*	13730	100	11720	400	224	63
	RGF 9-8-2	14235	100	12200	200	225	34
	RGF 9-13-3*†	14690	85	12620	220	229	36
	RGF 9-21-11	18240	140	15170	180	374	39
	RGF 9-24-4	18890	250	16020	420	337	81
	RGF 9-27-5*†	19000	70	16360	220	299	37
	RGF 9-32-4	20610	120	17230	280	416	53
	RGF 9-34-8*†	21980	130	18410	250	443	50
	RGF 12-30-2*†	30230	160	25870	410	547	84
	Tahiti						
	Ta-P6-10*	9565	20	8410	140	116	20
	$Ta-P6-11*$	9830	30	8790	120	99	17
	Ta-P6-12	9920	40	8800	120	110	17
	$Ta-P6-13*$	10205	30	8990	120	122	17
	Ta-P6-14+	10120	50	9065	100	100	15
	$Ta-P7-1*$	8520	25	7830	200	57	27
	$Ta-P7-2*$	9255	30	8170	180	108	25
	$Ta-P7-4$	10250	40	8970	140	131	20
	Ta-P7-5	10575	50	9330	140	125	21
$\neg \psi$	Ta-P7-6	10850	50	9550	140	132	21
	$Ta-P7-7*$	11005	12	9580	140	149	20
	Ta-P7-8	11280	30	9800	140	155	21
	Ta-P7-9 $Ta-P7-10$	11495	30	9980	140	160	21
	Ta-P7-11	11930 12875	50 40	10280 10830	140	177 233	22 22
	Ta-P7-12	12800	30	10800	140 160	226	25
	Ta-P7-13	12695	60	11010	160	179	25
	Ta-P7-14	12710	50	11090	160	170	24
	$Ta-P7-15$	12865	50	11030	160	201	25
	Ta-P7-16	12905	50	11090	160	198	25
	Ta-P7-17	13065	30	11430	200	171	29
	Ta-P7-18*	13465	40	11630	220	198	33
	Ta-P7-19*	13750	30	11790	220	216	34
	$Ta-P8-1\ddagger$	12905	30	11230	120	177	18
	Ta-P8-2‡	13335	30	11690	110	171	17
	Ta-P8-3‡	13665	35	12010	110	171	17
	BC =	11,715	$TNT(AL98=11707$				
					12310		
					$\widetilde{\Xi}^{\circ}$	¢,	

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TABLE 1. Comparison of Ages Obtained on Corals by AMS ¹⁴C and TIMS ²³⁰Th (*Continued*)

Note: All ages are expressed in yr before 1950 (BP) and statistical uncertainties are given at the 2σ level. ¹⁴C ages are conventional ages with a reservoir correction of 400 yr for Barbados and New Guinea and 300 the weighted mean and error (except for sample KWA-I-1, for which individual ¹⁴C ages are listed in italics). The U-Th ages of KWA-I-1 are from Dia et al. (1992). Sample Mu8-30-310.5 is composed of bothryoidal aragonite precipitated at shallow depth. All other samples are corals; species lists can be found in previous publications (Bard et al. 1990a, 1996). All samples were checked by XRD prior to dating to verify the absence of secondary low and/or high magnesium calcite $(<1%)$.

*Reported ²³⁰Th age is the weighted mean of 2 or more replicates (cf. Bard *et al.* 1993, 1996 for all individual ages). ^{\dagger}Reported ¹⁴C age is the weighted mean of 2 or more replicates (cf. Bard *et al.* 1993, 1996 for all individual ages). **‡Age was not reported previously.**

repeated measurements of NBS-010 (mean of $-7.0 \pm 0.7\%$ $2\sigma_m$, 29 measurements) and NBS-SRM960-NIST4321B (mean = $-35.6 \pm 1.5\%$ $2\sigma_m$, 6 meas.). The $\delta^{234}U_i$ values obtained on the Tahiti and Mururoa samples range between 140 and 150, with a mean value of $147 \pm 2\%$ (one standard deviation (SD) on 42 measurements). This average is very close to the values measured on presentday seawater (144 \pm 4‰ SD on 9 meas., Chen, Edwards and Wasserburg (1986)) and on modern and recent corals (145 ± 5‰, SD on 25 meas., Bard *et al.* (1990a); 150 ± 1‰, SD on 20 meas., Edwards et al. (1993); 148 ± 2‰, SD on 3 meas., Szabo et al. (1994); 149 ± 1‰, SD on 3 meas., Stirling et al. (1995)). This further confirms that the samples used for this study are devoid of diagenetic alteration and remained closed systems for U-Th in the past. In addition, the absence of secondary calcite \langle <1%) was also checked in triplicate by x-ray diffraction (XRD) in the samples selected for dating.

¹⁴C ages were measured by accelerator mass spectrometry (AMS) on the Tandetron facility installed at Gif-sur-Yvette (Arnold *et al.* 1987). 200–300 mg carbonate samples were first ground into millimeter-sized pieces preparatory to a strong acid leaching procedure (>40% weight loss) to remove surface contaminants (Bard et al. 1990b). Large carbonate subsamples $(15-18 \text{ mg})$ were then converted to CO_2 and reduced to graphite in order to produce at least two accelerator targets for most samples and hence to increase the ${}^{14}C$ precision. Each carbonate subsample was composed of several grains selected randomly, which should help to minimize the influence of intra-annual changes of the ¹⁴C reservoir ages, as shown by Brown *et al.* (1993). The ¹⁴C ages for Mururoa and Tahiti samples were corrected for 300 yr, which is a mean ¹⁴C reservoir age based on preanthropogenic data from the tropical South Pacific (Bard 1988). A reservoir age of 400 yr is used for corals from Barbados and New Guinea.

Fig. 1. AMS ¹⁴C ages plotted vs. TIMS ²³⁰Th ages obtained on corals. ¹⁴C ages are conventional ages in yr BP with statistical errors given at the 2 σ level. Δ = the data from New-Guinea (Edwards *et al.* 1993), $O =$ data from Barbados, \bullet = data from Tahiti and \bullet = the data from Mururoa. The thin wiggly curve is the smoothed tree-ring calibration and the thick solid line is the 1:1 line. For ages beyond the Younger Dryas/Preboreal boundary (10,000 BP) the coral data can be approximated by a simple linear equation: [cal BP] = $1.168 \times$ [¹⁴C age BP], or even better by a second-order polynomial: [cal BP] = -3.0126 \times 10⁻⁶ \times [¹⁴C age BP]² + 1.2896 \times [¹⁴C age BP] - 1005.

In addition to the samples collected by coring, we analyzed a very old *Porites lutea* sample collected in the lower uplifted terrace of Huon Peninsula, Papua New Guinea. This coral, sample KWA-I-1, was previously dated by TIMS U-Th at $41,100 \pm 500$ BP (Dia et al. 1992). Sample contamination and chemistry blank reproducibility are critical problems in dating such an old sample by ¹⁴C (see Bard et al. 1993 for blank measurements obtained on calcite and aragonite). Four different pieces of KWA-I-1 were dated (*i.e.*, 8 C-Fe targets) and the individual ¹⁴C ages are listed in Table 1 together with the weighted mean age based on these four values. The agreement among the four replicates is not optimal, which could be due to a small and residual contamination of this sample. The 14C age of KWA-I-1 remains tentative and more samples should be dated in the same time range to confirm its surprisingly high $\Delta^{14}C$ (ca. 700 %o).

Fig. 2. Blowup of the Figure 1 diagram between 7000 and 25,000 cal BP. Symbols as in Fig. 1.

RESULTS AND COMPARISON WITH PREVIOUS CALIBRATIONS

As previously shown, there is a large difference between the ¹⁴C and ²³⁰Th ages (Bard *et al.* 1990a). The magnitude of the $14C - 230T$ h age difference observed on the Tahiti and Mururoa samples (Figs. ¹and 2) agrees well with previous studies of corals (Bard et al. 1990a, 1993; Edwards et al. 1993). The accuracy of ²³⁰Th ages is further demonstrated by the excellent agreement with the recently revised dendrocalibration by Kromer and Spurk (1998), in particular in the critical range of the Ger- man pine calibration (10,000-11,900 cal BP; see Fig. 3).

Altogether, these two different calibration methods lead to the reconstruction of significant variations of the atmospheric ¹⁴C/¹²C ratio through time (Fig. 4). In particular, the new data from Mururoa confirm clearly that the atmospheric $\Delta^{14}C$ was ca. 400–500% higher at ca. 20,000–30,000 cal BP and that it has essentially decreased during the period between 18,000 and 7000 cal BP. This longterm Δ^{14} C decrease has been attributed to a concomitant long-term increase of the intensity of the geomagnetic field (Bard et al. 1990a; Bard 1997; Stuiver et al. 1991).

In the critical range between 9500 and 12,000 cal BP, the coral results are in agreement with the data obtained from varved sediments from Lake Gosciaz (Goslar et al. 1995), further confirming the new synchronization between the oak and the pine chronologies (Kromer and Spurk 1998). A new cali-

Fig. 3. Blowup of the Figure 2 diagram between 9500 and 13,500 cal BP. Symbols as in Fig. 1, except tree-ring calibration represented with gray error bars (from Kromer and Spurk 1998).

bration data set based on marine varves from the Cariaco Basin was recently proposed by Hughen et al. (1998). The Cariaco calibration curve goes back to 12,500 cal BP and is also in excellent agreement with our coral data (Hughen et al. 1998, especially Fig. 3b).

The coral ²³⁰Th ages together with the Gościąż and Cariaco varve data finally confirm that the other calibrations based on varved sediments from Sweden (Wohlfarth 1996), Holzmaar in Germany (Hajdas et al. 1995), and Soppensee in Switzerland (Hajdas et al. 1993) are still in error even after the recent additions of so-called "missing varves" (-900 "missing varves" were added to the chronology from Holzmaar (Hajdas et al. 1995); ~550 "missing varves" were added to the chronology from Soppensee (Hajdas et al. 1993); ~500 "missing varves" were added to the Swedish chronology (Wohlfarth 1996)).

An independent check on the coral ${}^{14}C-{}^{230}Th$ calibration can be obtained by analyzing volcanic ash layers that can be recognized and dated by counting annual couplets in Greenland ice cores ("cryovarves"). Grönvold et al. (1995) have identified and characterized chemically the Saksunar and Vedde ash layers, which occurred respectively at $10,180 \pm 120$ and $11,980 \pm 160$ cal BP, according to the GRIP core chronology (2σ errors). These two ash layers have recently been redated by AMS

Fig. 4. $\Delta^{14}C$ vs. time as calculated by using the AMS ¹⁴C ages vs. TIMS ²³⁰Th comparison. Statistical errors for coral data are provided at the 2 σ level. Symbols as in Fig. 1.

on terrestrial plant macrofossils at, respectively, 8960 ± 140 ¹⁴C yr BP (2σ error based on 3 AMS ¹⁴C ages from Birks *et al.* 1996) and 10,330 \pm 60¹⁴C yr BP (2 σ error based on 11 AMS ¹⁴C ages from Bard et al. 1994 and Birks et al. 1996). The data for these two volcanic events clearly demonstrate the compatibility of the German pine tree-ring chronology, 230 Th ages of corals, Gosciaz varves and GRIP annual counts in the time range around the Younger Dryas/Preboreal boundary dated at *ca*. 11,500 cal BP.

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