

²³⁰Th-²³⁴U AND ¹⁴C AGES OBTAINED BY MASS SPECTROMETRY ON CORALS

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INTRODUCTION

In 1988, Fairbanks conducted a drilling expedition off the south coast of Barbados to recover submerged corals contemporaneous with the last deglaciation. Core recovery was excellent and >30 different samples were dated by conventional β -counting techniques (Fairbanks 1989). At about the same time, we developed, at Lamont, the thermal ionization mass spectrometry (TIMS) technique to obtain precise U-Th ages (Edwards 1988), and to compare them with the ¹⁴C estimates measured on the same samples. A surprising result was that the discrepancy between ¹⁴C and U-Th ages increased through time to *ca.* 3000–3500 yr at *ca.* 15,000 ¹⁴C BP (Bard *et al.* 1990a). Because the three youngest samples yielded U-Th ages in agreement with their calibrated ¹⁴C ages, we concluded initially that the TIMS U-Th determinations were not only precise, but also accurate, and that the ¹⁴C vs. U-Th data set could be used for a first-order ¹⁴C calibration.

We replicated ¹⁴C measurements through accelerator mass spectrometry (AMS) on very small samples, which enabled us to use strong acid leaching to eliminate surface contaminants (see Bard *et al.* 1990b for technical procedures and the report of initial measurements). To establish global uniformity of the observed phenomenon, we also measured ages in new samples from very different environments, Mururoa Atoll and Isabela Island in the tropical Pacific Ocean.

RESULTS OBTAINED BY AMS AND TIMS ON BARBADOS CORALS

In five runs of the Gif-sur-Yvette Tandemtron, we obtained *ca.* 80 new ¹⁴C determinations on *ca.* 20 samples previously dated by β -counting (Fairbanks 1989, 1990) and TIMS (Bard *et al.* 1990a). The large quantity of replicates enables us to calculate weighted-mean AMS ¹⁴C ages with an improved precision (better than ± 200 yr at 2σ for the last 15,000 yr). The new AMS results agree to a first order with the β -counting estimates. However, we further confirm our previous observation (Bard *et al.* 1990b) that, for ages beyond the Holocene, the AMS determinations are often slightly older than the β -counting estimates (see Table 1A–C). The samples prepared for AMS were strongly leached with acid (loss of *ca.* 30–40% of the total weight), whereas the samples measured by β -counting were not (except samples RGF7-5-5, RGF7-16-2, RGF9-27-5 and RGF12-30-2). Consequently, a minor (<1pmC) contamination might cause this discrepancy. To illustrate how the calculation of $\Delta^{14}\text{C}$ is sensitive to old ages, we cite the deepest *A. palmata* sample (RGF 9-27-5), which was AMS-dated 3 times at Gif (weighted mean = 16,360 \pm 220 BP [2σ]), once at Arizona (16,145 \pm 240 BP [2σ], Fairbanks 1990) and twice by TIMS at Lamont (weighted mean = 19,000 \pm 70 BP [2σ]). Thus, the $\Delta^{14}\text{C}$ of this sample is *ca.* 300 ‰ instead of *ca.* 350 ‰ initially reported for that period, using β -counting ages obtained without leaching procedures.

Figure 1 shows results obtained by AMS and β -counting vs. the ages obtained by TIMS. Figure 2 shows enlarged detail of the intercomparison, which clearly illustrates the concordance between

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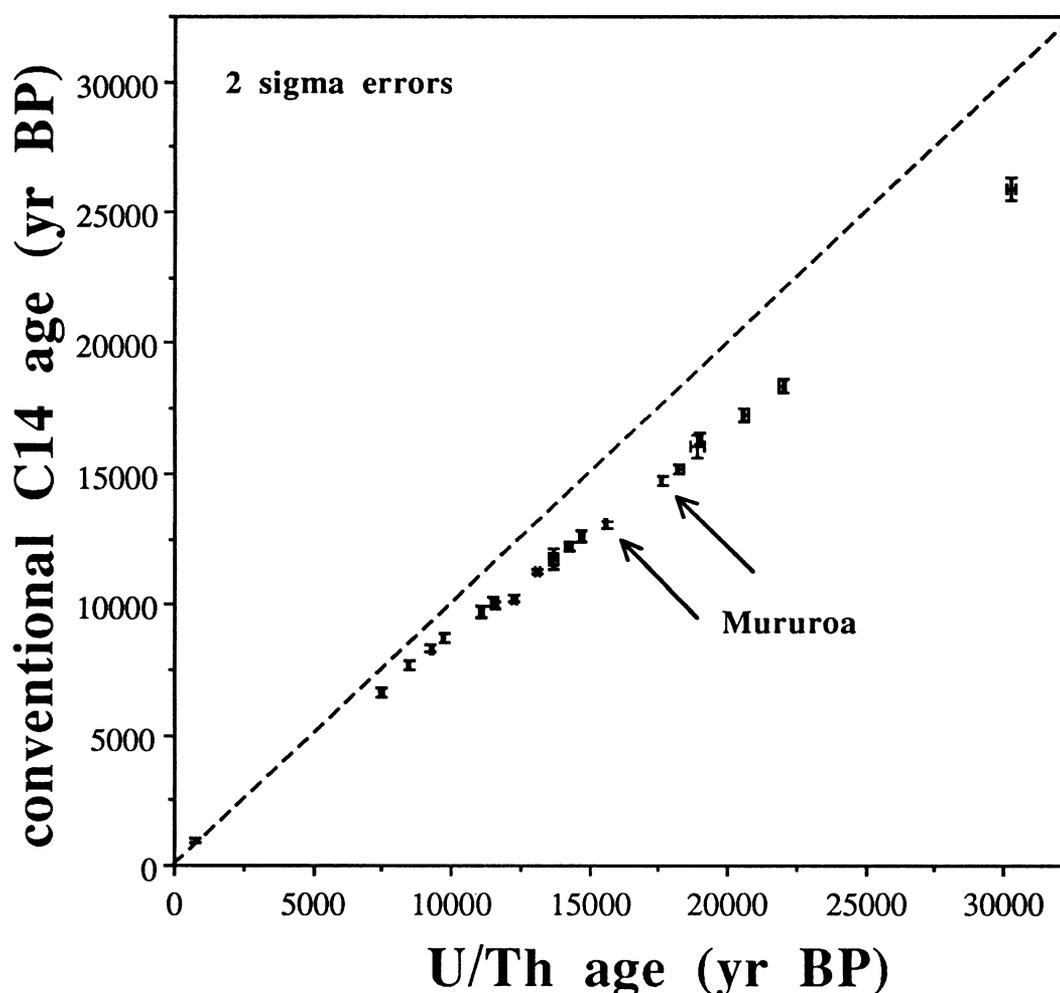


Fig. 1. U-Th ages obtained by TIMS plotted vs. AMS ^{14}C ages obtained on corals from Barbados and Mururoa. ^{14}C ages are conventional ages in yr BP with statistical errors given at 2σ , which does not include the error in the reservoir correction. For the interval between 8500 and 20,000 ^{14}C yr BP, a simple linear calibration is: cal age BP = $1.24(^{14}\text{C}$ age BP) - 840.

our reconstruction and the dendrochronological determinations of Kromer & Becker (1992).

We also studied the 400-yr reservoir age correction by dating a very recent Barbados coral drilled on the flat part of the reef at *ca.* 7 m below the present sea level (RGF B56). The U-Th age is 773 ± 10 BP [2σ] (weighted mean on 2 replicates); the ^{14}C age is 956 ± 90 BP [2σ] (weighted mean on 4 measurements), which leads to a calibrated age of 775–957 cal BP, using the “atmospheric” calibration program of the Quaternary Isotope Laboratory, University of Washington. This cal age is in statistical agreement with the U-Th estimate. However, we can also apply the marine ^{14}C calibration of the same program (with $\Delta R = 0 \pm 0$) on the raw ^{14}C age (1356 ± 90 BP [2σ]). In that case, the calibrated age is 787–967 cal yr BP which is close but slightly older than the U-Th estimate.

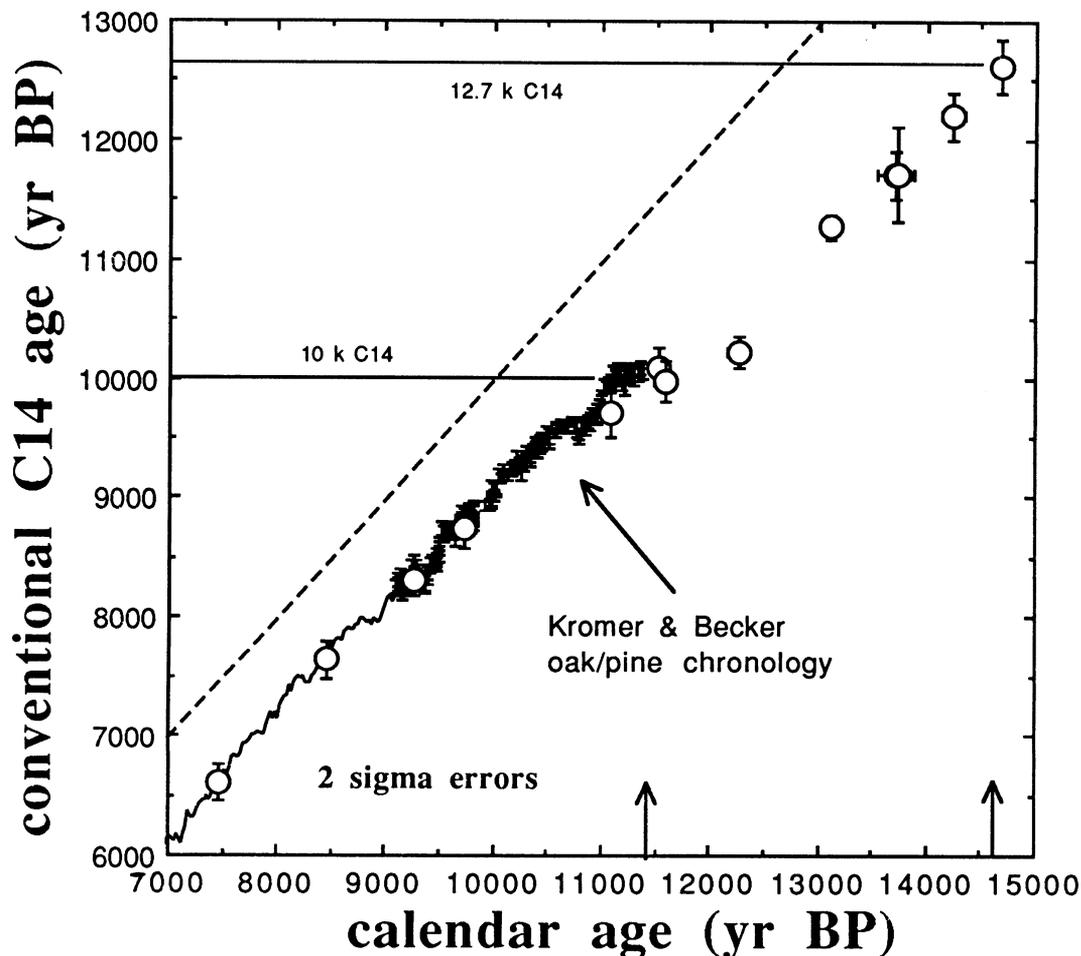


Fig. 2. Enlargement of Figure 1, also showing the tree-ring data base. Until 9100 cal BP, the solid curve is the 20-yr running average of the dendrochronology. Between 9100 and 11,400 cal BP, it comprises German oak and pine data (Kromer & Becker 1992). All errors are given at 2σ (for ^{14}C ages of corals they do not include the error in the reservoir correction).

Thus, the reservoir age offshore Barbados is not very different from our adopted value of 400 yr ($\text{DIC } \Delta^{14}\text{C} \approx -50\text{‰}$), which is a mean for low- and mid-latitude open ocean waters (Bard 1988). However, if we take the results obtained on sample RGF B56 at face value, we could also adopt a different reservoir correction. Indeed, there are still some uncertainties about the pre-anthropogenic $\Delta^{14}\text{C}$ of the West Atlantic/Caribbean zone; Druffel & Suess (1983) reported $\Delta^{14}\text{C}$ values of -50‰ for Belize and Florida corals while Nozaki *et al.* (1978) obtained $\Delta^{14}\text{C}$ values of *ca.* -30‰ (≈ 250 yr) for 19th-century annual bands collected from a Bermuda coral head. Perfect agreement should not be expected, and the reservoir age probably exhibits small temporal variations in response to changes of tradewind, temperature and mixed-layer depth (Bard 1988).

RESULTS OBTAINED BY AMS AND TIMS ON MURUROA CORALS

We measured ages of two coral specimens collected with a land-based deviated drill ($\approx 30^\circ$) at *ca.* 170 m below present sea level, offshore Mururoa atoll, French Polynesia. For sample 313, the U-Th age is $17,595 \pm 70$ BP [2σ] (weighted mean on 3 replicates), whereas the ^{14}C age is $14,735 \pm$

TABLE 1A. Comparison of ages obtained by classical ^{14}C β counting, ^{14}C -AMS and U-Th-TIMS. All ages are expressed as yr BP (before 1950). The ^{14}C ages are conventional ages with a reservoir correction of 400 yr. Statistical errors are given at 2σ , which does not include the error in reservoir correction.

Sample	U/Th (yr BP)	\pm 2 σ	C14 (yr BP) L-DGO	\pm 2 σ	C14 (yr BP) Geochron	\pm 2 σ	Leaching *	Replication **	C14 (yr BP) Gif-sur-Yvette	\pm 2 σ
Galapagos 2#1	283	13								
Galapagos 2#2	265	6								
Galapagos 1#1	295	20								
Galapagos 1#2	269	12								
RGF B56#1	773	11					+		825	180
RGF B56#2	773	28					+	du	1050	180
RGF B56#3							+		995	160
RGF B56#4							+	du	975	180
RGF7-4-2#1	7460	80	6400	200			+		6640	200
RGF7-4-2#2							+	du	6560	220
RGF7-5-5#1	8450	50			7780	220	+		7640	240
RGF7-5-5#2							+	du	7640	220
RGF7-12-2#1	9285	95	8200	200			+		8160	220
RGF7-12-2#2	9250	80					+	du	8080	360
RGF7-12-2#3							+		8410	220
RGF7-12-2#4							+	du	8410	220
RGF7-16-2#1	9730	50			9050	250	+		8750	240
RGF7-16-2#2							+	du	8750	240
RGF7-27-4#1	11090	70	9400	200			+		9690	320
RGF7-27-4#2							+	du	9720	280
RGF12-5-2#1	11590	60	10100	200			+		9760	380
RGF12-5-2#2							+		10070	340
RGF12-5-2#3							+	du	10020	240
RGF12-6-7#1	11530	70	9800	200			+		9730	400
RGF12-6-7#2							+		10080	240
RGF12-6-7#3							+	du	10250	260
RGF12-9-5#1	12260	90	10300	200			+		10320	320
RGF12-9-5#2							+	du	10280	280
RGF12-9-5#3							+		10280	260
RGF12-9-5#4							+	du	10090	220

*+ = strong leaching (>30% weight loss of the sample; for AMS samples only)

**Results obtained by duplicating (du) or triplicating (tri) the AMS runs on 2 or 3 aliquots of the same Fe+C powder.

150 BP [2σ] (weighted mean on 5 age determinations). For sample 315, the U-Th age is $15,585 \pm 50$ yr BP [2σ] (weighted mean on 3 replicates), whereas the ^{14}C age is $13,060 \pm 140$ yr BP [2σ] (weighted mean on 5 age determinations). These new results show again a significant difference between the two geochronometers, corresponding to $\Delta^{14}\text{C}$ values of 330 ± 30 ‰ and 290 ± 25 ‰ at *ca.* 18,000 and 16,000 Th yr BP, respectively. These values are in excellent concordance with AMS redeterminations of the Barbados ^{14}C ages (*cf.* Fig. 1). This new comparative data set of U-series and ^{14}C ages demonstrates that the large discrepancies first evidenced in Barbados are not caused by local alteration processes.

TABLE 1B. Comparison of ages obtained by classical ¹⁴C β counting, ¹⁴C-AMS and U-Th-TIMS. All ages are expressed as yr BP (before 1950). The ¹⁴C ages are conventional ages with a reservoir correction of 400 yr. Statistical errors are given at 2 σ, which does not include the error in reservoir correction.

Sample	U/Th (yr BP)	± 2σ	C14 (yr BP) L-DGO	± 2σ	C14 (yr BP) Geochron	± 2σ	Leaching *	Replication **	C14 (yr BP) Gif-sur-Yvette	± 2σ	C14 (yr BP) other AMS labs	± 2σ
RGF12-16-5#1	13220	110	10900	200			+		11640	280		
RGF12-16-5#2	12970	120					+		10940	260		
RGF12-16-5#3							+		11020	240		
RGF12-16-5#4							+		11200	260		
RGF12-16-5#5							+	du	11490	280		
RGF12-16-5#6							+		11570	340		
RGF12-16-5#7							+	du	11190	260		
RGF12-21-6#1	13700	170	11850	200			+		11630	260		
RGF12-21-6#2							+	du	11820	300		
RGF12-21-10#1	13800	140	11800	200			+		11720	400		
RGF12-21-10#2	13660	140										
RGF9-8-2#1	14235	100	11800	400			+		12240	260		
RGF9-8-2#2							+	du	12130	320		
RGF9-13-3#1	14660	160	12500	200			+		12000	420		
RGF9-13-3#2	14700	100					+		12910	340		
RGF9-13-3#3							+	du	12710	380		
RGF9-21-11#1	18240	140	14700	400					14930	400		
RGF9-21-11#2									15390	400		
RGF9-21-11#3							+		15110	320		
RGF9-21-11#4							+	du	15230	340		
RGF9-24-04	18890	250	15400	400			+		16020	420		
RGF9-27-5#1	18980	90			17085	520	+		16260	420	16145	240
RGF9-27-5#2	19030	100					+		16240	360	AMS Arizona	
RGF9-27-5#3							+	du	16550	360		
RGF9-27-4			15200	520							15850	240
RGF9-32-4#1	20610	120	16700	600			+		16920	420	AMS Arizona	
RGF9-32-4#2							+	du	17470	380		
RGF9-34-8#1	21930	150	18200	400			+		18820	520		
RGF9-34-8#2	22130	260					+		18260	420		
RGF9-34-8#3							+	du	18300	400		

*+ = strong leaching (>30% weight loss of the sample; for AMS samples only).

**Results which have been obtained by duplicating (du) or triplicating (tri) the AMS runs on 2 or 3 aliquots of the same Fe+C powder.

RESULTS OBTAINED BY TIMS ON GALAPAGOS CORALS

To further study the reliability of TIMS age estimates on very young samples, we dated samples from a *Pavona clavus* colony collected on Isabela Island (Galapagos). This recent coral head was killed in 1954 due to tectonic uplift from the shelf of Urvina Bay. A high-resolution chronology was previously obtained for this colony through counting of growth bands imaged by x-rays (Shen *et al.* 1991 and references therein). We dated two samples by TIMS: Gal. 2, which has a sclerochronological age of 283–285 cal BP, and yielded a U-Th age of 269 ± 6 BP [2 σ] (weighted mean on 2 replicates), and Gal. 1, dated at 296–298 cal BP with a U-Th age of 277 ± 10 BP [2 σ] (weighted mean on 2 replicates). Thus, the results obtained by TIMS are very close to the true ages, but not in complete agreement if the statistical errors are taken into account.

We made these first attempts to date very young corals on a rather old solid-source mass spectrometer (VG-MM30) equipped with an analog Daly detector. For these analyses, the ²³⁰Th+ ion currents were very close to background, and it would have been preferable to make additional

TABLE 1C. Comparison of ages obtained by classical ^{14}C β counting, ^{14}C -AMS and U-Th-TIMS. All ages are expressed as yr BP (before 1950). The ^{14}C ages are conventional ages with a reservoir correction of 400 yr. Statistical errors are given at 2σ , which does not include the error in reservoir correction.

Sample	U/Th (yr BP)	$\pm 2\sigma$	C14 (yr BP) Geochron	$\pm 2\sigma$	Leaching *	Replication **	C14 (yr BP) Gif-sur-Yvette	$\pm 2\sigma$	C14 (yr BP) other AMS labs AMS Zürich	$\pm 2\sigma$
RGF12-30-2#1	30470	240	27120	1520	+		26290	860	26480	1000
RGF12-30-2#2	30040	210					24600	820		
RGF12-30-2#3							25290	840		
RGF12-30-2#4					+		25790	840		
RGF12-30-2#5					+	du	25370	660		
RGF1-17-4#1	70820	600	30080	3520	+		>48000			
RGF1-17-4#2							>48000			
RGF8-27-1#1	79400	700	36240	7980						
RGF8-27-1#2	77800	800								
Mururoa 315#1	15550	100					13160	280		
Mururoa 315#2	15660	90					13160	300		
Mururoa 315#3	15550	80			+		13030	360		
Mururoa 315#4					+		12840	360		
Mururoa 315#5					+	du	12990	300		
Mururoa 313#1	17500	120					14690	320		
Mururoa 313#2	17630	100					14770	340		
Mururoa 313#3	17650	130			+		14630	360		
Mururoa 313#4					+		14620	360		
Mururoa 313#5					+	du	14910	320		
Mururoa Irène 30#1	259000	6000			+		>48700			
Mururoa Irène 30#2					+	du	>48600			
Mururoa Irène 30#3					+	tri	>48400			
Mururoa Irène 30#4					+		>48500			
Ir30 0.25 mg					+		>46700			
Ir30 0.22 mg					+		>47200			
Ir30 0.19 mg					+		>47900			
Ir30 0.13 mg					+		>39700			
stalagmite					+		>48300			
IAEA-C1 marble					+		>48300			

*+ = strong leaching (>30% weight loss of the sample; for AMS samples only).

**Results obtained by duplicating (du) or triplicating (tri) the AMS runs on 2 or 3 aliquots of the same Fe+C powder.

¹Quantification of the blank was conducted on three samples: 1) a stalagmite from Orgnac cave (>300 ka by α -counting U-Th dating); 2) the IAEA-C1 marble; and 3) sample Irène-30 dated by TIMS at 259 ± 6 ka. The ^{14}C contamination was also tested on very small aliquots of Irène-30 (0.25 mg C–0.13 mg C). Note that the normal size of the coral samples is always between 1 and 2 mg C. A more comprehensive study of the blank levels on carbonate and organic samples is under preparation and will be published elsewhere (Bard & Arnold, ms. in preparation).

baseline corrections at mass 229.5 and 230.5, instead of using the simpler procedure of measuring a common baseline at mass 227.5. However, this would have caused significant loss in ^{230}Th measurement time, which would have reduced measurement precision. Consequently, we believe that these preliminary results constitute a trade-off between accuracy and precision. Ideally, we will replicate the analyses using the VG Sector-54-30 mass spectrometer recently installed at LGE-Marseille; this instrument is equipped with an ion-counting Daly detector that can decrease significantly the detection limit (by about 100 times) for small ion currents. Another explanation for the 10–20 yr discrepancy could also come from the sclerochronological estimates. It could be caused by problems in correlating different coral sections which had to be corrected for small gaps (Shen *et al.* 1991), or to additional stress bands in response to exceptionally cold years (see, *e.g.*, Druffel & Linick 1978, who showed evidence for three stress bands in a 30-yr section of *Montastera annularis* from the Florida Keys).

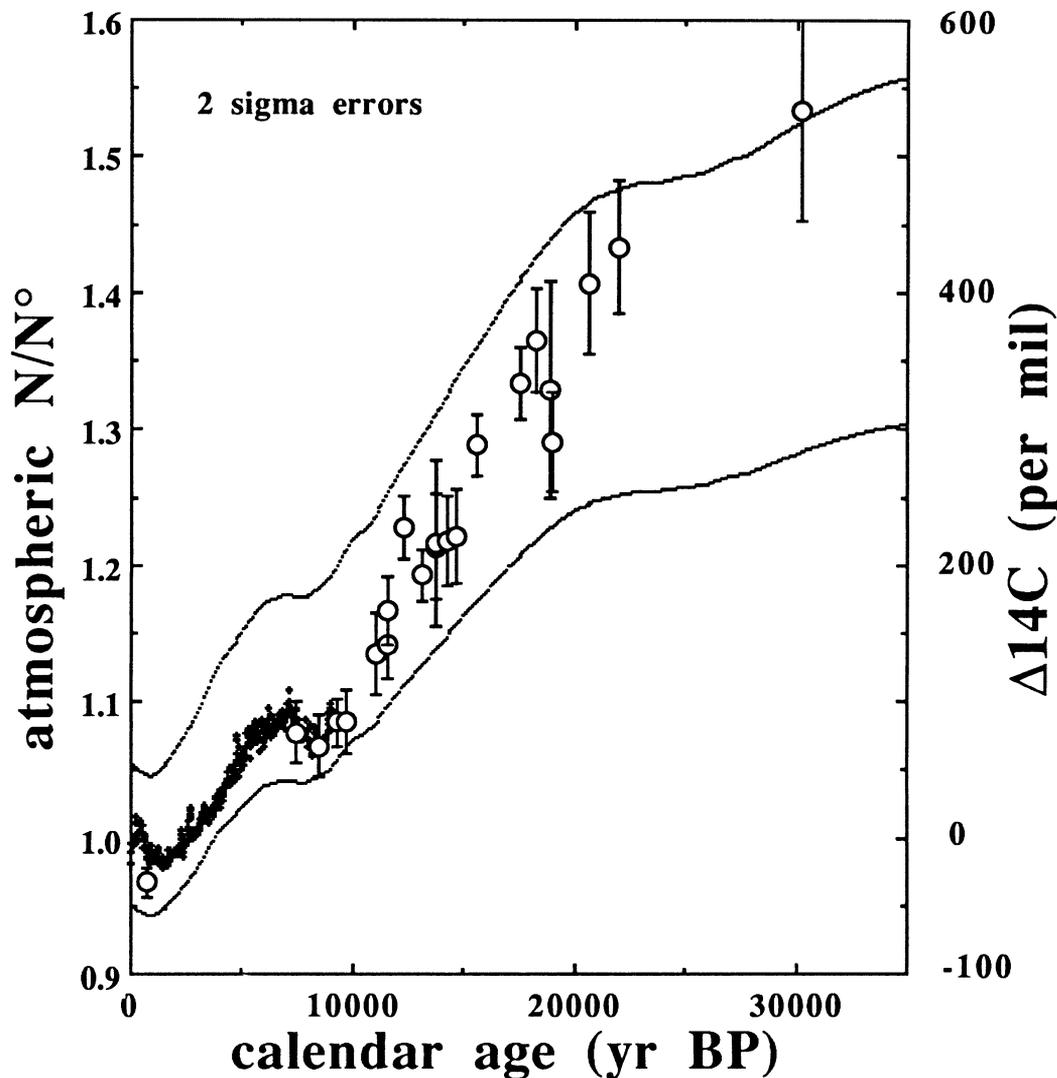


Fig. 3. $\Delta^{14}\text{C}$ vs. time as calculated by using the AMS- ^{14}C vs. U-Th comparison. + = dendrochronological calibration. All statistical errors are quoted at 2σ . For sample RGF12-30-2, we used the ^{14}C replicates obtained by AMS after very strong acid-leaching procedures. The two sets of solid lines correspond to the envelopes of $\Delta^{14}\text{C}$ expected as a response to geomagnetic field variations as reconstructed by McElhinny & Senanayake (1982). For those calculations, we used a two-box model and assumed constant errors of $\pm 10\%$ of the present-day dipole (Bard *et al.* 1990a).

DISCUSSION

The new AMS and TIMS results presented here confirm the precision and accuracy of the U-Th geochronometer applied to scleratinian corals. We show again that the ^{14}C ages are systematically younger than true ages during most of the ^{14}C application range (except for the brief period between 500 and 2500 cal BP). For the 10,000–30,000 ^{14}C yr BP period, other authors found similar discrepancies between ^{14}C and K-Ar dating (Gillot & Cornette 1986), between ^{14}C and thermoluminescence (TL) dating (Valladas & Valladas 1987; Bell 1991) and between ^{14}C ages and U-Th ages obtained in samples from speleothems (Vogel 1983) or corals (Veeh & Veevers 1970; Moore, Normak & Szabo 1990).

On the basis of available data, it is clear that the calendar duration of the Holocene chronozone (0–10,000 ^{14}C yr BP) ranges from 11,200 to 11,500 cal yr, and that the Younger Dryas chronozone (10,000–10,800 ^{14}C yr BP or 10,000–11,000 ^{14}C yr BP) corresponds to *ca.* 1000–1600 cal yr. These results agree with the study of varved sediments from Lake Gosciarz (Rozanski *et al.* 1992; Goslar *et al.* 1992).

If we assume that the ^2H and ^{13}C records measured by Becker, Kromer and Trimborn (1991) on German pine sections represents the whole European climate, we estimate an age of *ca.* 9700 ^{14}C yr BP for the midpoint of the Younger Dryas/Preboreal ^2H – ^{13}C transition. Consequently, the U-Th age of the midpoint can be dated at about 11,100 yr by means of the U-Th vs. ^{14}C age comparison (close to sample RGF 7-27-4).

The AMS- ^{14}C age of the early Bølling $\delta^{18}\text{O}$ shift varies between 12,700 BP (Ammann & Lotter 1989) and 12,500 BP (Bard *et al.* 1987), which corresponds to U-Th ages between 14,700 and 14,500 BP. As proposed by Broecker (1992), this benchmark could be used to correlate records obtained from ice cores, tree rings, corals, oceanic and continental sediments, but we should also expect a correlation problem since 12,700 ^{14}C yr BP corresponds to another ^{14}C age plateau (Ammann & Lotter 1989).

Bard *et al.* (1990a) and Stuiver *et al.* (1991) discuss in detail the geophysical interpretation of the difference observed between ^{14}C and U-Th ages obtained in corals. As Figure 3 shows, the results can be explained mainly by changes in cosmogenic nuclide production linked to the gradual decrease of the geomagnetic field, which has been documented by paleomagnetism studies (Barbetti & Flude 1979; McElhinny & Senanayake 1982; Mazaud *et al.* 1991; Salis & Bonhommet 1992). Changes in the carbon cycle also can account for *ca.* 10–20% of the age discrepancy observed between the two geochronometers; these may involve fluctuations in deep-ocean ventilation (Shackleton *et al.* 1988; Broecker *et al.* 1990) and variations in the carbon content of the atmosphere (Bernier, Oeschger & Stauffer 1980; Delmas, Ascencio & Legrand 1980) and biosphere (Adams *et al.* 1990).

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