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^{14}C AGES OF OSTRACODES FROM PLEISTOCENE LAKE SEDIMENTS OF THE WESTERN GREAT BASIN, USA—RESULTS OF PROGRESSIVE ACID LEACHING

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ABSTRACT. Progressive dissolution experiments were performed on samples of ostracode shells from lacustrine sediments from the western Great Basin to remove contamination of the surface by secondary calcite. The observed age differences between the external and residual fractions were as great as 2000 to 6000 yr. A "plateau" in ages of the last fractions was obtained only for 1 sample; however, results of repeated experiments resulted in very good agreement of the final ages. A comparison with previously published chronologies based on bulk radiocarbon ages of ostracodes from Wilson Creek (Benson et al. 1990) shows that leaching is imperative for dating samples older than 20 ka BP. This study focuses on the problem of contamination and its removal. However, the final chronology of the Wilson Creek Formation (and other late Pleistocene lacustrine sediments) will require additional dating of other sections as well as establishment of a reservoir effect correction.

INTRODUCTION

The Great Basin (Figure 1) is a system of closed basin lakes located west of the Continental Divide, enclosed by mountains (the Sierras, the Cascades, the Wasatch Plateau) and characterized by semi-arid to arid climate. The region appears to have been very sensitive to Pleistocene climate change and the Late Glacial Maximum lake levels were uniformly high.

It was the wet/dry cycles, which are visible as ancient playa or shorelines of once deep lakes, that caught the attention of geologists. The first radiocarbon results were obtained by Libby in 1955 on samples from sediments of Searles Lake, California (Libby 1955). Research that followed established a pattern and timing of climatic oscillations in the Great Basin region and correlation with other regions such as the North Atlantic. It appears from studies by Benson et al. (1998) that rapid climatic events—perhaps equivalent to the Dansgaard/Oeschger events observed in the Greenland ice cores (GRIP and GISP2) and Heinrich events (HE) manifested by layers of ice-rafted debris in the North Atlantic (Bond et al. 1997)—might have an imprint in wet/dry cycles in the Great Basin region.

Reliable chronologies are critical for reconstructions of the past climate. Patterns of climatic changes require correlation and synchronization between regions and records. Correlations proposed by Benson et al. (1998) imply synchrony between the North Atlantic and Great Basin climatic cycles. For example, the last 2 low-stands of Lake Russell (Mono Lake) appear to correlate with HE1 (13.8 ka BP) and HE2 (21 ka BP). However, correlation of the older HE is difficult. This might be caused by chronological problems, namely contamination of the ostracodes with modern carbon and an unknown correction for reservoir effect. Kent et al. (2002) recognized the modern carbon contamination issue and they assumed that the residual carbonate measurement yielded a maximum estimate of the original ^{14}C in the carbonate (minimum apparent age). They also recognized that the $^{40}Ar/^{39}Ar$ ash chronology is complicated and that minimum relative sanidine results yield a maximum age. Further, they considered it likely that the large magnetic excursion in the Wilson Creek Formation, known as the "Mono Lake Excursion," was equivalent to the "Laschamp" geomagnetic

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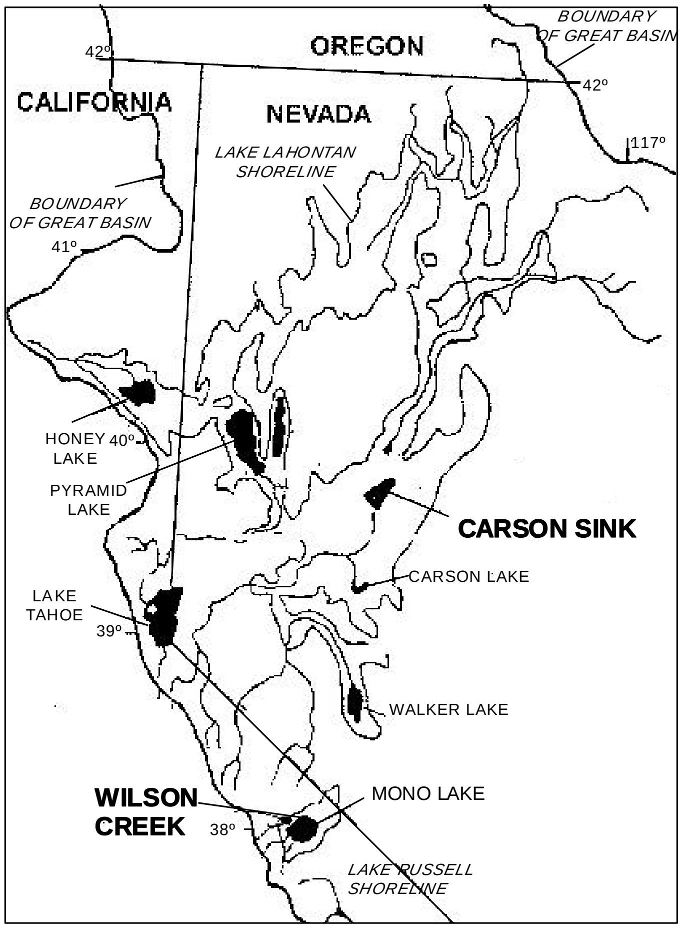

Figure 1 Map of the Great Basin adopted from Benson et al. (1990) showing location of studied sites Mono Lake and Wilson Creek Formation (California) and Carson Sink (Nevada).

excursion. Implications of such assessments are far reaching for paleoclimatic correlations, as well as correlation among paleomagnetic intensity records of the last 40 to 50 ka BP.

An alternative correlation to that of Kent et al. (2002) has already been proposed by Benson et al. (1998, 2003). Benson et al. (2003) rebutted the possibility of contamination with modern carbon and suggested an extremely large reservoir correction in order to bring the residual ^{14}C results reported by Kent et al. (2002) back into agreement with their previous assessment (Benson et al. 1998). However, such a procedure would have to be applied to both sets of data and the offset would remain unchanged. Benson et al. (2003) showed that Ash #15 of the Wilson Creek Formation has a similar composite to the ash that has been identified at Carson Sink (coincidently, here we report ostracode data associated with that ash). A chemical match between ashes allows but does not require them correlated; thus, this issue remains unresolved.

Our study focuses on one important aspect of these issues: the ^{14}C chronology of the Wilson Creek Formation and problems connected to the possible contamination by secondary calcite deposited on the surface of ostracode shell selected from the sediments of this section.

SURFACE CONTAMINATION, DIFFUSION, AND SECONDARY CALCITE

Concerns about the possibility of contamination of carbonate samples with younger carbon pose a challenge to ^{14}C dating. One mechanism that causes contamination is young carbon dioxide diffusing through the porous surface into the core of the carbonate sample. The effects of surface contamination by modern CO_2 and diffusion into the body of the sample have been estimated and measured by Broecker and Orr (1958), who dated tufa from the Pyramid and Searles lakes. They found that, although the effect is on the order of 700 yr for a 20,000-yr-old sample, it might be avoided by leaching 80% of the surface material. In the same publication, the authors described a possibility of contamination by secondary calcite as unlikely given the arid climatic conditions in the Great Basin region. On the other hand, treatment similar to that proposed by Broecker and Orr (1958) should be sufficient for removal of younger secondary carbon. Combined ^{14}C and U/Th chronologies from Lahontan showed the effect on ages of tufa from the last high-stand of the lake, 13 ka BP (Lin et al. 1998). Burr et al. (1992) showed that 80% leaching of coral surfaces provided satisfactory ^{14}C ages.

Ostracode shells have a high surface to volume ratio; therefore, surface contamination should be taken into consideration as a potential source of error. In this study, we show results of leaching ostracodes from 2 locations in the Great Basin and the effect this procedure has on the ^{14}C chronology.

RESERVOIR EFFECT

Studies of aquatic environments require that a reservoir effect is accounted for, in addition to the problems of contamination with modern carbon. A site-specific correction is required that depends on the input of carbon-depleted water (rivers, springs), evaporation to precipitation, lake surface to volume ratios, and factors which control gas exchange (Broecker and Orr 1958; Broecker and Walton 1959). These factors can be estimated, but often they are subjected to temporal fluctuations such as lake-level variability. Moreover, due to hydrothermal inputs of ^{14}C-free CO_2, the apparent ages of Mono Lake water might be as high as 6300 yr (Broecker et al. 1988). ^{14}C activities measured for contemporary terrestrial deposits or the ^{14}C age of water can give estimates of reservoir effect. Yet, the concern of this study is the possibility of contamination with "modern carbon," i.e., ages being too young. Our goals outlined in this study are to obtain ages of ostracodes which are free of such contamination and to build a reliable estimate of the initial ^{14}C that was incorporated into ostracode shells when formed.

^{14}C DATING OF OSTRACODES FROM CARSON SINK AND WILSON CREEK

The Sites

The section of the Wilson Creek Formation from its type locality along Wilson Creek is located at the north shore of Mono Lake, California (38°N, 118°W). The Wilson Creek Formation contains lacustrine sediments that were deposited in Lake Russell, the extended paleolake that existed during the last glacial cycle. Nineteen tephra layers found in 7-m-thick deposits of sediments can be correlated around the basin. An anomalous paleomagnetic secular variation was found at ash layer #15 and called the Mono Lake Excursion (Liddicoat 1996; Liddicoat and Coe 1979).

The Carson Sink stratigraphic section is an artificial cut exposed in the west bank of Carson River, ~20 km north east of Fallon, Nevada (39°N, 118°W). This section contains lacustrine sediments with layers composed of almost 100% ostracodes. Two white volcanic ash layers are present: the Wono ash layer and about 50 cm below the Carson Sink bed. As reported by Benson et al. (2003), the Carson Sink bed and ash layer #15 in the Wilson Creek Formation have a nearly identical chemical composition.

The Method

Sediment samples were disaggregated in deionized water and sieved. Ostracodes were hand-picked from the >250-mm fraction. Dating of fractions released in progressive leaching requires a large amount of material and, where possible, 100 mg of ostracodes were picked. However, as this is very tedious and time-consuming work, most of the samples contained 50–80 mg of ostracodes. Samples were placed in the "thumb" part of the acidification flask with 20 mL of concentrated (80%) phosphoric acid in the main tube (Figure 2). After a vacuum of 10^{-4} mb was achieved, the reaction flask was closed and the sample was mixed with the acid and left to react at 70 °C. The pressure of CO_2 released during acidification was sporadically monitored and gas was frozen in a storage tube when the amount was sufficient for 1 AMS ^{14}C sample (about 1–2 mg of C, which is based upon a 12% yield of C from of $CaCO_3$). As each split was collected, the remaining material was left to react and the CO_2 was collected repeatedly for dating in the same manner described above. The variation and limits on sample size weight dictated the number of fractions collected for each sample. Additionally, the number of fractions measured varied due to the sporadic measurements of the pressure, which were based on the time of the reaction. The collected CO_2 was reduced to graphite in a reaction with H_2 over cobalt at 625 °C (Vogel et al. 1984). Samples were measured at the ETH/PSI accelerator mass spectrometry (AMS) facility following the procedure described by Bonani et al. (1987). Conventional ^{14}C ages were calculated according to the protocol of Stuiver and Polach (1977).

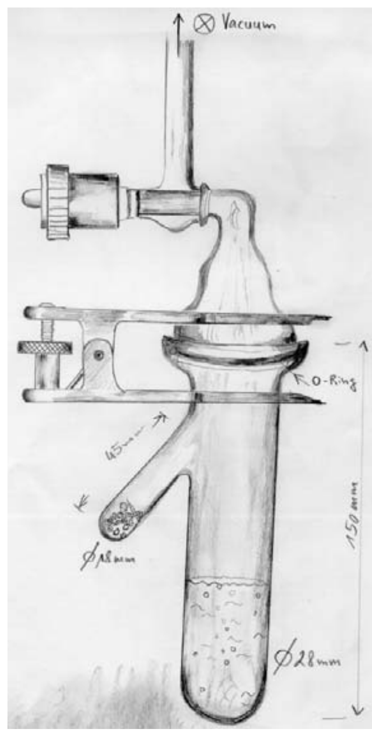

Figure 2 Attachable acidification flask used for dissolution of carbonate samples. Samples are placed in a "thumb" of the lower part using a long funnel. Acid is poured into the main chamber using an acid dispenser. The upper part with an o-ring is placed on the top and fixed with a clip. The whole chamber is attached to the vacuum and graphitization system.

RESULTS AND DISCUSSION

In the first stage of the study, several experiments were performed on ostracodes from Carson Sink. Conventional ^{14}C ages of these ostracodes are listed in Table 1. Duplicate experiments were done for 2 levels in an attempt to leach as much as possible of the ostracode shells. Each of the duplicate

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experiments were performed on samples that contained at least 15 mg of ostracodes. The differences in ^{14}C ages caused by successive leaching are shown in Figure 3a and Figure 3b.

Table 1 Results of progressive leaching of ostracode shells from Carson Sink. Conventional ^{14}C ages are quoted with 1-σ error. The fraction on which age measurement was performed is based on pressure of CO_2 released during consecutive leaching steps, where '1' stands for the whole sample.

Lab nr ETH-	Sample nr	Height (cm)[a]	Fraction measured	^{14}C age (BP)	$\delta^{13}C$ (‰)	Weight (mg)
19682	CS59	–59	0–0.7	24,310 ± 190	–1.2 ± 1.2	56
			0.7–1.0	27,250 ± 220	–0.8 ± 1.2	
19682[b]	CS59/2	–59	0–0.2	22,660 ± 160	1.6 ± 1.2	103.7
			0.2–0.31	27,180 ± 250	1.2 ± 1.2	
			0.31–1.0	27,830 ± 240	4.1 ± 1.2	
19680	CS3	–3	0–0.64	21,930 ± 160	–0.2 ± 1.2	100
			0.64–0.76	24,060 ± 180	1.1 ± 1.2	
			0.76–1.0	25,680 ± 200	–0.3 ± 1.2	
WONO	ASH	0				
19681	CS19	+19	0–0.23	24,630 ± 180	–1.6 ± 1.2	82
			0.23–0.47	27,540 ± 230	–2.0 ± 1.2	
			0.47–1.0	28,070 ± 240	–1.2 ± 1.2	
19681[b]	CS19/2	+19	0–0.8	22,020 ± 180	1.3 ± 1.2	100.8
			0.08–0.21	26,120 ± 230	1.0 ± 1.2	
			0.28–0.32	25,000 ± 340	0.9 ± 1.2	
			0.32–1.0	28,130 ± 250	1.2 ± 1.2	

[a]Stratigraphic position (from bottom to top) in relation to the Wono ash layer, here at 0 cm; heights below the Wono layer are shown as negative values and heights above the layer are shown as positive values.
[b]Repeat measurement on the rest material.

As well as determining the extent of exogenous carbon contamination with respect to post-leach age determinations, there were interesting results from the dating of successive leaches. The age offset between the external fraction and the residual fraction varies depending on the percentage of material in each fraction dated. For example, the largest difference is observed for sample CS19/2 (Figure 3a), where the difference between first (external) fraction and the residue is ~6000 yr. The same sample leached in the first experiment, CS19, shows a smaller difference (~3400 yr). We also observed that smaller fractions of the first leach returned the youngest ^{14}C ages in samples large enough for duplicate experiments. For example, first fractions from samples CS59 and 59/2, a 70% first leach was 24,310 ± 190 BP and a 20% first leach was only 22,660 ± 160 BP, respectively. This is shown again with CS19 and 19/2, where a 23% leach was 24,610 ± 190 BP and an 8% leach was 22,020 ± 180 BP, respectively. However, the ages for the final leaches in CS19 and CS19/2 are in very good agreement (28,099 ± 173 BP, $\chi^2 = 0.03$). Final ages obtained by leaching the samples CS59 and CS59/2 (Figure 3b) agree within the 2-σ range, despite the differences in ages of the first fractions (27,515 ± 290 BP, $\chi^2 = 3.17$). Other observed differences in ^{14}C ages between successive fractions could be associated with the size of the fraction. One fraction of CS19/2 was very small (less than 0.5 mg C, compared to all the other fractions, 1.5–2 mg of C), which might be the reason for the slightly lower age in the preceding fraction. Nevertheless, these 2 ages are in agreement in the 2-σ range.

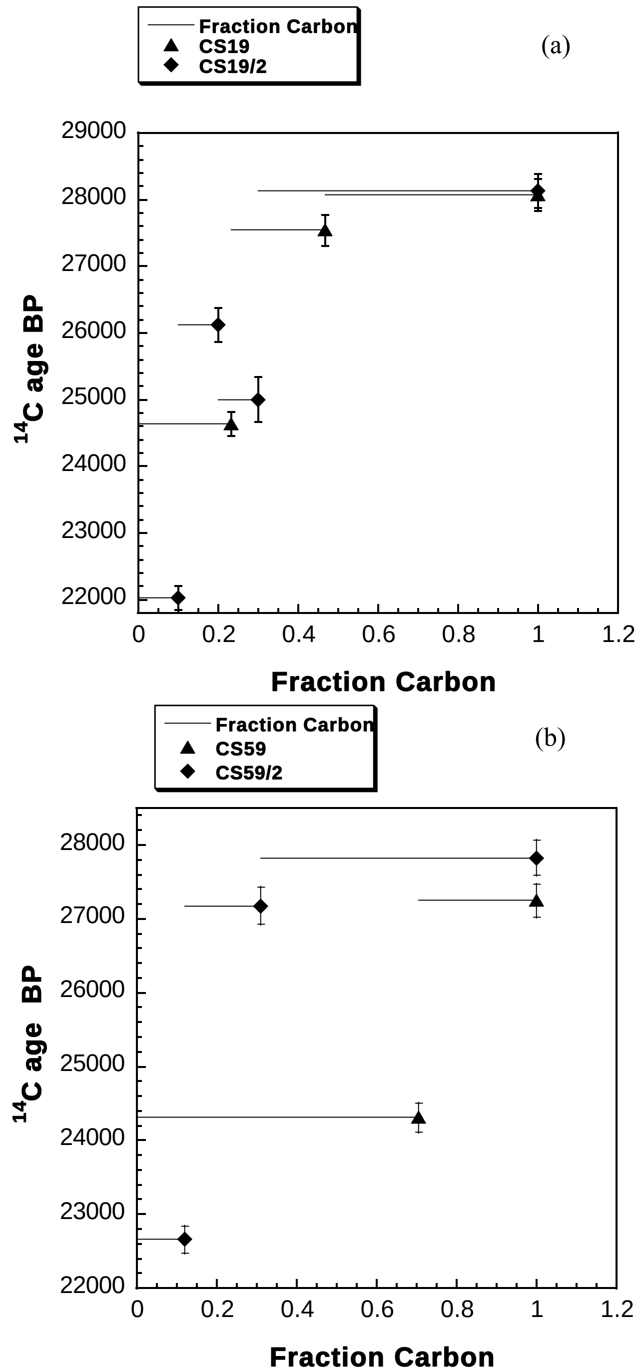

Figure 3 Results of leaching ostracodes from Carson Sink. ^{14}C ages obtained on consecutive fractions of carbon (based on pressure of CO_2 released during leaching steps) are plotted together with 1-σ error for samples (a) CS19 (ETH-19681) and (b) CS59 (ETH-19682). Triangles show the first round of measurements and diamonds show results of the second progressive leaching of ostracodes.

Results obtained on ostracodes from the Wilson Creek Formation are listed in Table 2 and plotted in Figures 4a,b. Ostracodes from 13 levels above the formation base were dated using the leaching process outlined in the Methods section. Leaching experiments were first carried out on 3 levels above the base of the section: 50 cm, 100 cm, and 160 cm. Total samples available for the 50-cm level allowed us to run 2 successive leaching experiments (WC50, WC50/2) to check on the reproducibility of results, with each leaching fraction producing sufficient carbon for analysis. The difference between the first fraction leached and the final age of the final fraction in both experiments is as high as 6000 yr. The final ages of inside fractions for WC50 and WC50/2 are within the 2-σ range (mean weighted value = 40,533 ± 940 BP, $\chi^2 = 2.5$). In both experiments, the first leach of 35% of the sample is ~35,500 BP. Moreover, the second 37% leach and the remaining 27% of residue fraction appeared to plateau in age (Figure 4a).

Table 2 Results of progressive leaching of ostracode shells from Wilson Creek. Conventional ^{14}C ages are quoted with 1-σ error. The sample number corresponds to the height given in cm. The fraction on which age measurement was performed is based on the pressure of CO_2 released during consecutive leaching steps, where '1' stands for the whole sample.

Lab nr ETH-	Sample nr	Height (cm)[a]	Fraction measured	^{14}C age (BP)	$\delta^{13}C$ (‰)	Weight (mg)
19889	WC50	50	0–0.36	35,500 ± 530	0.7 ± 1.2	110.0
			0.36–0.73	39,450 ± 660	2.4 ± 1.2	
			0.73–1.0	39,700 ± 790[b]	2.6 ± 1.2	
20298	WC50/2	50	0–0.37	35,710 ± 510	1.7 ± 1.2	77.0
			0.37–1.0	41,590 ± 890[b]	2.0 ± 1.2	
21056	WC51	51	0.41–1.0	46,100 ± 1700[b]	0.7 ± 1.2	69.0
21057	WC61	61	0.43–1.0	39,200 ± 710	1.8 ± 1.2	46.0
21057[c]	WC61/2	61	0–1.0	37,820 ± 550	6.1 ± 1.2	27.5
21059	WC81	81	0.62–1.0	39,800 ± 730	0.4 ± 1.2	107.0
21060	WC91	91	0.56–1.0	35,810 ± 500	1.1 ± 1.2	62.0
21060[c]	WC91/2	91	0–0.47	33,680 ± 370	3.4 ± 1.2	46.1
			0.47–1.0	34,600 ± 400	7.5 ± 1.2	
20190	WC100	100	0.5–0.74	31,910 ± 380[b]	–0.6 ± 1.2	55.4
	WC100	100	0.74–1.0	36,250 ± 430[b]	3.4 ± 1.2	
21061	WC102	102	0.27–1.0	38,080 ± 620	2.1 ± 1.2	90.0
21061[c]	WC102/2	102	0.34–1.0	39,890 ± 690	6.4 ± 1.2	84.4
21062	WC112	112	0.36–1.0	34,490 ± 440	–1.0 ± 1.2	73.0
21062[c]	WC112/2	112	0–0.25	34,950 ± 440	6.6 ± 1.2	78.3
			0.25–1.0	35,660 ± 450	6.4 ± 1.2	
21063	WC122	122	0.43–1.0	31,270 ± 330	0.0 ± 1.2	77.0
21063[c]	WC122/2	122	0–0.52	31,920 ± 390	4.5 ± 1.2	37.9
			0.52–1.0	32,130 ± 320	7.3 ± 1.2	
21064	WC132	132	0.48–1.0	32,800 ± 380	–1.2 ± 1.2	90.0
21064[c]	WC132/2	132	0–1.0	32,910 ± 350	7.5 ± 1.2	17.7
21065	WC142	142	0.39–1.0	33,770 ± 410	–1.0 ± 1.2	66.0
20191	WC160	160	0.5–0.77	31,470 ± 340	1.9 ± 1.2	63.9
			0.77–1.0	33,610 ± 360[b]	3.0 ± 1.2	
21067	WC163	163	0–0.5	31,140 ± 330	0.2 ± 1.2	40.3
			0.5–1.0	32,700 ± 380	0.3 ± 1.2	

[a]Height above the formation base.
[b]Ages published by Kent et al. (2002).
[c]Repeat measurement on the rest material.

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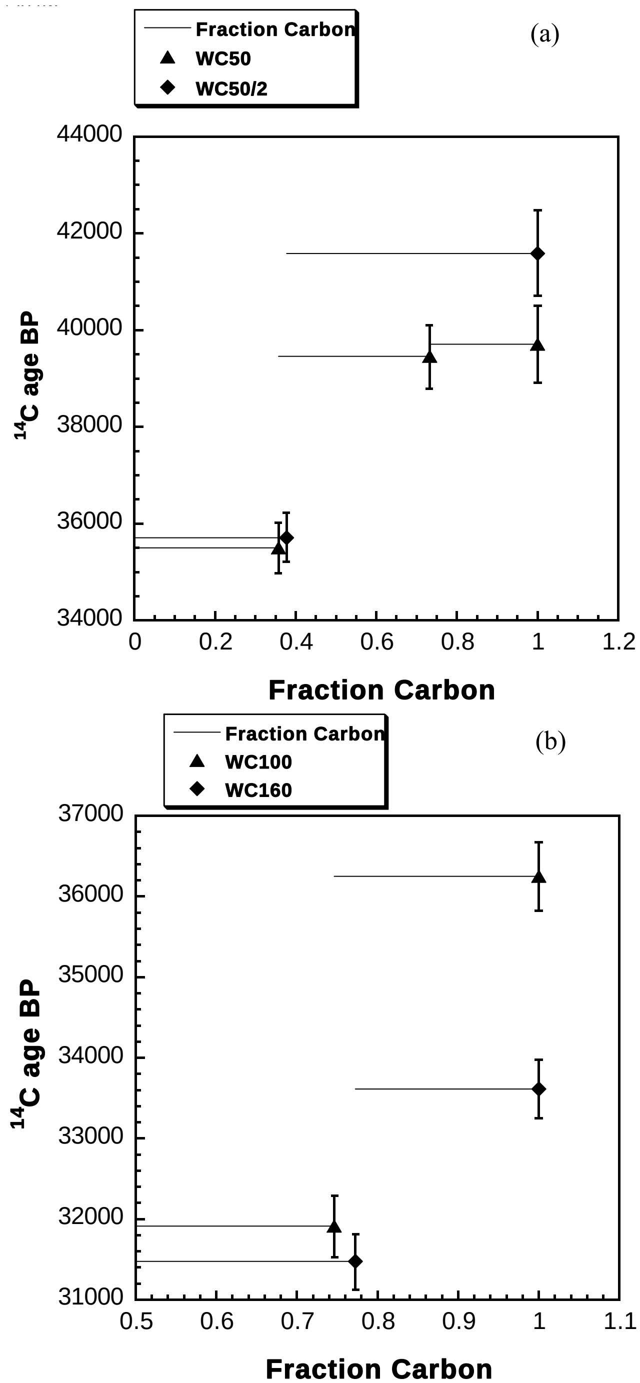

Figure 4 Results of leaching ostracodes from the Wilson Creek Formation. (a): ages obtained on fractions from 2 samples from the 50-cm level are shown as triangles (WC50, ETH-19889) and diamonds (WC50/2, ETH-20298); (b): leaching experiment was performed on 50% pre-leached samples from level 100 cm (ETH-20190) (squares) and 160 cm (ETH-20191) (diamonds).

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Samples from the 100-cm and 160-cm levels had been leached (50%) prior to our "online" leaching. In these samples, the first fraction is younger than the residual fraction by ~4000 yr (Figure 4b). All of the levels listed in Table 2 were analyzed in 1999. We have recently performed our leaching experiments and analysis on some of the residual fractions of samples that contained a sufficient amount of C (Table 2, samples marked by footnote *c*). Residual fraction samples were processed and subjected to our leaching experiments (as described in the Methods section) and submitted for AMS analysis if a minimum of 1 mg carbon was collected. In general, the external fractions are younger than the residual fraction; however, the second round of measurements indicates a need for stronger than 30–50% removal of the surface as shown by the difference of ~1000 yr between ages of the both inside fractions obtained for samples WC112 and WC122 (Figure 5).

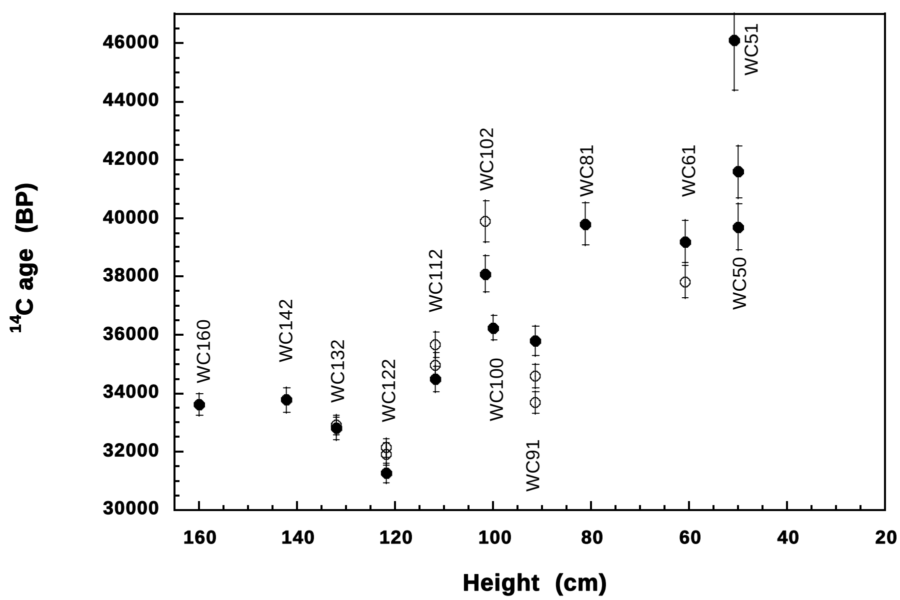

Figure 5 ^{14}C chronology of Wilson Creek. Filled circles show results from the first round of measurements. Results from repeated measurements are shown as open circles with corresponding sample number. In each of the repeated pairs, the older age corresponds to the "inside" fraction. Samples WC61 and WC132 were dated as whole shell.

Two of the samples (WC61/2 and WC132/2) were processed as a whole because they contained only 27.50 and 17.7 mg of ostracodes, respectively, which were insufficient for 2 and more fractions. The age obtained for WC61/2 was younger by ~1500 yr compared to the residual fraction measured in the first round of measurements. However, this effect is not observed for the younger sample WC132/2, which turned out to be the same age as the residual fraction of WC132.

DISCUSSION

^{14}C Chronology of the Wilson Creek Formation

We undertook the present study to address the question of the reliability of ostracode shell dating in studies such as those for the Wilson Creek Formation. A final chronology of lacustrine sediments deposited during the last 40,000 yr in the Wilson Creek Formation is urgently needed for the purpose of correlating palaeoclimatic records of the Great Basin with other regions such as the North Atlantic (Benson et al. 1998). Additionally, the timing of the paleomagnetic excursion found in the Wilson Creek Formation must be resolved to allow proper correlation of paleomagnetic records (Benson et al. 2003; Kent et al. 2002).

Most of the ^{14}C ages of ostracode shells from the Wilson Creek Formation published by Kent et al. (2002) have been obtained in the younger part of the section. In that work, the ostracodes and tufa nodules were leached prior to the dating so that at least 40% of the surface was removed. In this study, we investigated the older part of the section in detail. We also applied progressive leaching and duplicated those experiments to check reproducibility where sample sizes allowed, showing that various stages of leaching do, in fact, produce different ^{14}C ages, and that the extent of the leach is an important factor for adequate removal of "modern/younger" carbon contamination. As we have shown, age differences obtained in progressive leaching of the samples underscore the need for 50 to 75% leaching of ostracode shells prior to ^{14}C dating (Figure 5).

Developing the ^{14}C chronology of this record will require determining the true reservoir correction for the section. Because the best estimates vary between 1500 and 6000 yr (Broecker et al. 1988), estimation of the reservoir correction will require additional research, such as dating terrestrial (reservoir-free) records, and correlation with the Wilson Creek Formation using tephra layers. However, the extent of the possible offsets caused by contamination with secondary calcite does not require knowledge of the reservoir effect and can be determined by comparing ages measured on whole ostracode shells with results from leached ostracode samples from the same section. In Figure 6, ^{14}C ages of leached ostracode shells (this study and Kent et al. 2002) and a chronology based on whole shells (Benson et al. 1990) are plotted for comparison. Although resolution of the dating is still not sufficient, the offset between both chronologies in the oldest parts of the section are up to

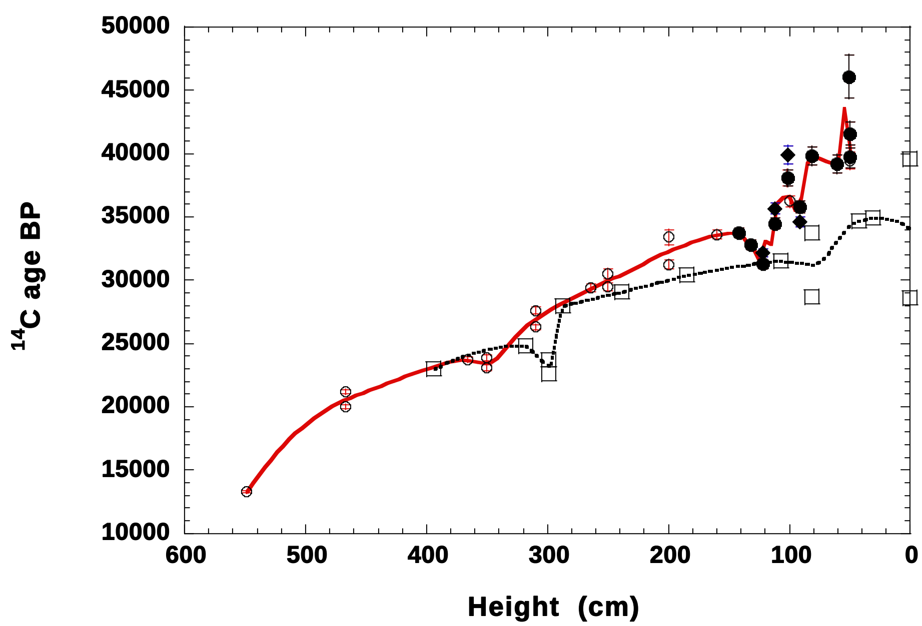

Figure 6 Comparison between chronologies based on leached ostracodes (filled circles and diamonds, this study; open circles show ages obtained by Kent et al. [2002]) and ages obtained on whole shell (open squares) by Benson et al. (1990).

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6000+ yr. We also note that the younger intervals appear to agree quite well, which points to contamination becoming a significant factor for the very old samples. Benson et al. (2003) suggested that ages published by Kent et al. (2002) may have been obtained on reworked shells that were too old. Such a possibility cannot be entirely ruled out. However, there are 2 points to consider which support the Kent et al. (2002) older chronology. The first is that, as we have observed, the chronologies agree quite well in the younger part, implying that both studies used similar methods in selecting material. The second consideration is that the results of our present study show that there can be significant differences in the apparent ^{14}C ages of successive leached fractions of ostracode shells, and that another explanation for the divergence in ages between Benson et al. (1990) and Kent et al. (2002) may be due to exogenous carbon effects in untreated shells.

CONCLUSIONS

Progressive leaching of ostracode shells from Carson Sink and Wilson Creek, Mono Lake (Great Basin) resulted in older ^{14}C ages of the final fraction of progressively leached samples. Differences of up to 6000 yr between ages of the external and the residual fractions have been observed in these experiments, suggesting that the extent of leaching as a pretreatment for these samples is an important factor. Our procedure of leaching 80% of the shell improved the chronology of the record, although we concede that contamination could extend beyond this fraction and produce anomalous ages.

In samples from the oldest section of the Wilson Creek segment (+20,000 BP), the chronology based on our analysis of leached ostracodes returned ages older (by up to 6000 yr) than the previously established ^{14}C chronology of Benson et al. (1990) based on a whole shell measurements, and corroborated Kent et al. (2002) results obtained from similarly leached shell samples.

The differences between various chronologies of the region call for extensive studies that would establish a final ^{14}C chronology. This study presents a step towards such an improved chronology of the Wilson Creek Formation.

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