

## DATING OF LATE PLEISTOCENE TREE-RING SERIES FROM JAPAN

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**ABSTRACT.** We have radiocarbon dated series of tree rings from 2 fossil trees (named ND-113 and the Fuji tree) buried in fossil volcanic avalanche deposits in Japan. They are dendrochronologically floating, dating beyond the tree-ring part of the  $^{14}\text{C}$  calibration curve. The trees show about 350 and 400 annual rings, respectively, which are dated in intervals of 2 to 10 yr. Both sequences are wiggle-matched to the calibration curve IntCal09. This resulted in an age range of 16,534–16,204 cal BP for ND-113, and 23,678–23,290 cal BP for the Fuji tree.

### INTRODUCTION

For the construction of atmospheric calibration curves for radiocarbon, tree rings form the most important archives (e.g. Baillie 1982). This is particularly true for tree rings absolutely dated by dendrochronology. Since the early work by Suess (1970), Stuiver and Kra (1986), and others, calibration of the  $^{14}\text{C}$  timescale based on dendrochronology has come a long way. The presently recommended calibration curve IntCal09 (Reimer et al. 2009) contains tree-ring data back to around 12,500 cal BP.

Beyond this dendro limit, the IntCal09 curve is based on marine data (Reimer et al. 2009). For tree rings, there is the promise of kauri wood from New Zealand, with a potential significant extension of the denro part of the IntCal curve (Balter 2006).

Today, tree-ring sequences from the Late Pleistocene do exist, including some that are dated by  $^{14}\text{C}$  (e.g. Kromer et al. 2004). This is also the case for the above-mentioned kauri wood (Turney et al. 2007). However, these sequences are not dated absolutely by dendrochronology, and are therefore floating chronologies.

Here, we report on 2 such floating chronologies from Japan, dated by  $^{14}\text{C}$ . One of the trees lived about 3.5 centuries, the other 4. The results are matched to the marine-derived part of the calibration curve IntCal09 to provide an absolute timescale. We selected these trees from Japan because they were available and they span a long time, which is relatively unique and potentially contain century-scale wiggles.

### LOCATION

Two Japanese boreal conifer samples of the Late Glacial period were prepared for  $^{14}\text{C}$  dating. The Fuji tree is a Japanese cypress (*Chamaecyparis obtuse*), found in 1969 on a hillside ( $35^{\circ}13'\text{N}$ ,  $138^{\circ}37'\text{E}$ ) near Mount Fuji, in the central part of Honshu Island, Japan. The tree shows ~400 annual rings with parts of the core and the sapwood lost. The tree was buried in a mud flow from the old Fuji volcano. The outer part (30 rings) of the tree has been  $^{14}\text{C}$  dated before to  $18,900 \pm 300$  BP by liquid scintillation counting (Fukuhura and Wada 1997).

The tree ND-113 is a tree of the genus *Picea* from a fossil forest, found in 1998 at the Oyazawa-Noda archaeological site ( $40^{\circ}47'\text{N}$ ,  $140^{\circ}40'\text{E}$ ) in the northeastern part of Honshu Island, Japan. This tree shows ~350 annual rings, with the bark being conserved. It was found under a 1-m-thick layer

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of pyroclastic flow deposit from the Towada Volcano. The eruption that formed this pyroclastic flow deposit was a huge one with an estimated Volcanic Explosivity Index (VEI) of 6.7 (Hayakawa 1985), leaving a large caldera lake. An accelerator mass spectrometry (AMS) date for this event was reported recently as 13.1 ka BP (Horiuchi et al. 2007).

The location of the trees in Japan is shown in Figure 1. A section of one of the trees, ND-113, is shown in Figure 2.



Figure 1 Geographical locations of the trees Fuji and ND-113 in Japan



Figure 2 Section of the ND-113 tree from Honshu Island, Japan

## METHODOLOGY

Wood samples were measured by both AMS and the conventional method (proportional gas counting) in Groningen (laboratory code GrA and GrN, respectively). The method chosen (AMS or conventional) was done solely on the basis of the amount of wood sample available.

The ND-113 tree was divided into 34 decadal tree-ring blocks. These samples were all large enough to be measured conventionally. The wood was pretreated by the standard AAA (acid-alkali-acid) method, combusted into pure CO<sub>2</sub>, and subsequently dated by proportional gas counting (Mook and Streurman 1983). The Fuji tree shows small rings, requiring <sup>14</sup>C dating by AMS for all samples from this tree. Originally, the Fuji wood was cut into 40 decadal tree-ring blocks. Later, additional blocks with higher temporal resolution (2 and 5 yr) were prepared.

All AMS samples were pretreated by AAA. After the chemical pretreatment, the samples were combusted and turned into CO<sub>2</sub> by an elemental analyzer (EA), coupled on-line with a stable isotope mass spectrometer. Next, the CO<sub>2</sub> was reduced to graphite by reacting under excess H<sub>2</sub> gas (Aerts-Bijma et al. 2001). The graphite was pressed into target holders, which are placed in the ion source of the AMS. The Groningen AMS facility is based on a 2.5MV Tandetron accelerator, and measures the <sup>14</sup>C concentration in the graphite (van der Plicht et al. 2000). The <sup>14</sup>C dates are reported in conventional yr BP, which includes correction by isotope fractionation using the <sup>13</sup>C isotope. For both the conventional and AMS dates, the δ<sup>13</sup>C values are measured by isotope ratio mass spectrometry (IRMS).

## RESULTS AND ANALYSIS

By fitting the data sets to the calibration graph, an absolute date can be obtained for the records. This is done by “wiggle-matching,” i.e. matching the data—which is a floating chronology—to the calibration curve, to form the absolute chronology. The wiggle-matching has been performed using the computer code OxCal version 4.1 (Bronk Ramsey 2001) with the IntCal 09 calibration curve (Reimer et al. 2009). The OxCal program uses Bayesian statistics to obtain the best fit, and also calculates the statistical uncertainty of the match.

The Fuji tree comprises close to 400 annual rings. Originally, decadal samples were taken; later on, more detailed studies were done based on 2-, 3-, or 5-yr temporal resolution. This “zooming in” was done when there were indications for wiggles. It is practically impossible to analyze all samples with a 2-yr temporal resolution. The results show good comparability within measurement error.

For almost all rings, duplicate samples were analyzed. In addition, some AMS runs were repeated in order to obtain better statistics. The measured data are averaged; results are shown in Table 1. The table shows the GrA numbers, sample name, rings analyzed, (averaged) <sup>14</sup>C age and error (1 σ) in yr BP, and the δ<sup>13</sup>C value. The last column shows the result of the wiggle-matching: the calibrated age obtained by OxCal in cal BP. The Fuji tree was determined to have lived from 23,678 to 23,290 cal BP. The matching error is 16 calendar years (1 σ).

Table 1 <sup>14</sup>C measurements for the Fuji tree.

GrA	Name	Rings	<sup>14</sup> C (yr BP)	σ	δ <sup>13</sup> C (‰)	cal BP
21474,83647	A1	1–10	19,300	65	-21.78	23,290
26404,26475	A2	11–20	19,450	65	-21.60	23,300
26405,26476	A3	21–30	19,375	65	-22.15	23,310
26406,26477	A4	31–40	19,320	60	-21.72	23,320
26407,26479	A5	41–50	19,305	60	-22.25	23,330

Table 1  $^{14}\text{C}$  measurements for the Fuji tree. (*Continued*)

GrA	Name	Rings	$^{14}\text{C}$ (yr BP)	$\sigma$	$\delta^{13}\text{C}$ (‰)	cal BP
26409,26481	A6	51–60	19,445	65	-22.09	23,340
26410,26482	A7	61–70	19,380	65	-22.04	23,350
26411,26483	A8	71–80	19,500	65	-21.42	23,360
26415,26485	A9	81–90	19,450	65	-21.39	23,370
26486,26889	A10	91–100	19,450	60	-21.12	23,380
33218,33733	C1	101–105	19,327	40	-20.98	23,388
26413,26487	A11	101–110	19,440	70	-20.93	23,390
33219,33735	C2	106–110	19,407	40	-21.55	23,393
33220,33736	C3	111–115	19,435	40	-21.25	23,398
26427,26489	A12	111–120	19,645	65	-21.40	23,400
33221,33734	C4	116–120	19,547	40	-21.32	23,403
33223,33738	C5	121–125	19,390	40	-21.24	23,408
26426,26490	A13	121–130	19,595	65	-21.55	23,410
33224,33739	C6	126–130	19,475	40	-21.27	23,413
33225,33740	C7	131–135	19,525	40	-21.13	23,418
26424,26492	A14	131–140	19,665	65	-21.50	23,420
33228,33753	C8	136–140	19,560	40	-21.35	23,423
33229,33754	C9	141–145	19,400	40	-21.47	23,428
26423,26493	A15	141–150	19,555	65	-21.69	23,430
33230,33755	C10	146–150	19,632	40	-21.69	23,433
33231	C11	151–155	19,505	55	-21.54	23,438
26421,26495	A16	151–160	19,650	50	-21.80	23,440
33233,33758	C12	156–160	19,607	40	-21.82	23,443
33234,33759	C13	161–165	19,565	40	-21.52	23,448
26419,26496	A17	161–170	19,785	65	-21.72	23,450
33235,33760	C14	166–170	19,627	40	-21.78	23,453
33238,33762	C15	171–175	19,500	40	-21.39	23,458
26417,26497	A18	171–180	19,735	65	-21.61	23,460
33239,33782	C16	176–180	19,612	40	-21.91	23,463
33240	C17	181–185	19,490	55	-21.58	23,468
26429,26499	A19	181–190	19,715	65	-21.94	23,470
33241,33784	C18	186–190	19,577	40	-21.95	23,473
33243,33785	C19	191–195	19,652	40	-21.43	23,478
26430,26500	A20	191–200	19,715	65	-21.90	23,480
33244,33743	C20	196–200	19,502	40	-21.96	23,483
33245,33764	C21	201–205	19,567	40	-21.68	23,488
26431,26564	A21	201–210	19,635	65	-22.08	23,490
33247,33765	C22	206–210	19,580	40	-21.18	23,493
33249,33766	C23	211–215	19,600	40	-21.88	23,498
26433,26570	A22	211–220	19,590	60	-22.06	23,500
33250,33768	C24	216–217	19,627	40	-21.62	23,502
29236,29263	B1	219–220	19,667	45	-21.53	23,504
29223,29265	B2	221–222	19,700	50	-21.70	23,506
29268,29224	B3	222–223	19,715	55	-22.01	23,508
26420,26565	A23	221–230	19,815	65	-21.62	23,510
29225,29269	B4	225–226	19,923	55	-21.92	23,510
29228,29271	B5	227–228	19,926	50	-21.83	23,512
29229	B6	229–230	19,670	95	-22.13	23,514
29237,29261	B7	231–232	19,782	45	-21.97	23,516
29227,29272	B8	233–234	19,696	55	-22.30	23,518

Table 1  $^{14}\text{C}$  measurements for the Fuji tree. (Continued)

GrA	Name	Rings	$^{14}\text{C}$ (yr BP)	$\sigma$	$\delta^{13}\text{C}$ (‰)	cal BP
26450,26566	A24	231–240	19,655	60	-22.38	23,518
29238,29273	B9	235–236	19,857	45	-22.31	23,520
29239,29274	B10	237–238	19,757	45	-22.42	23,522
29241,29276	B11	239–240	19,842	45	-22.65	23,524
29242,29278	B12	241–243	19,852	45	-22.65	23,527
29248,29279	B13	244–245	19,775	45	-22.17	23,529
26451,26568	A25	241–250	19,650	60	-22.02	23,530
29249,29281	B14	246–247	19,842	45	-22.14	23,531
29251,29281	B15	248–249	19,815	45	-22.15	23,533
29252,29283	B16	250–251	19,720	45	-22.06	23,535
29253,29331	B17	252–254	19,643	50	-22.36	23,538
29243,29294	B18	255–256	19,763	55	-22.80	23,538
29246,29295	B19	257–258	19,763	50	-22.01	23,540
29247,29296	B20	259–260	19,763	50	-22.33	23,542
29256,29297	B21	261–262	19,643	55	-22.51	23,544
29257,29299	B22	263–264	19,790	50	-22.04	23,546
26453,26563	A27	261–270	19,630	60	-21.85	23,548
29258,29300	B23	265–266	19,873	55	-21.94	23,548
29259,29301	B24	267–268	19,796	55	-22.32	23,550
29262,29304	B25	269–270	19,816	55	-22.24	23,552
33251,33769	C22	271–275	19,660	45	-22.92	23,556
26455,26573	A28	271–280	19,665	60	-22.51	23,558
33253,33744	C23	276–280	19,647	40	-21.95	23,561
33745	C24	281–285	19,730	90	-22.15	23,566
26456,26574	A29	285	19,760	60	-22.34	23,568
33746	C25	286–290	19,590	55	-21.87	23,571
33256,33770	C26	291–295	19,660	40	-21.11	23,576
26457,26575	A30	291–300	19,835	65	-22.30	23,578
33258,33771	C27	296–300	19,580	40	-22.04	23,581
33260,33773	C28	301–305	19,802	40	-21.76	23,586
26460,26576	A31	301–310	19,765	65	-22.50	23,588
33261,33775	C29	306–310	19,797	40	-21.43	23,591
33263	C30	311–315	19,770	55	-21.50	23,596
33264,33778	C31	316–320	19,745	40	-21.51	23,601
33265,33780	C32	321–325	19,720	40	-21.36	23,606
26462,26579	A33	321–330	19,915	60	-22.37	23,608
33267,33748	C33	326–330	19,740	40	-21.64	23,611
33268,33749	C34	331–335	19,745	40	-21.48	23,616
26463,26580	A34	331–340	19,880	65	-21.56	23,618
33269,33750	C35	336–340	19,665	40	-21.34	23,621
33270,33779	C36	341–345	19,730	40	-21.69	23,626
26461,26578	A32	341–350	19,795	65	-22.05	23,628
26465,26583	A35	341–350	19,700	60	-21.86	23,628
26466,26584	A36	351–360	19,755	60	-21.87	23,638
26467,26585	A37	361–370	19,815	60	-21.88	23,648
26470,26586	A38	371–380	19,760	65	-21.61	23,658
26471,26588	A39	381–390	19,760	65	-21.78	23,668
26472,26589	A40	391–400	19,675	65	-21.94	23,678

The ND-113 tree was dated conventionally. The ~350 rings were sampled with decadal resolution, yielding 34 samples. The results are shown in Table 2. The table shows the GrN number, sample name, rings analyzed,  $^{14}\text{C}$  age and error ( $1\sigma$ ) in yr BP, and the  $\delta^{13}\text{C}$  value. The precision varies depending on sample size, requiring measurement in different proportional counters. The last column shows the result of the wiggle-matching: the calibrated age obtained by OxCal in cal BP. The ND-113 tree was determined to have lived from 16,534 to 16,204 cal BP. The matching error is 44 calendar yr ( $1\sigma$ ).

Table 2  $^{14}\text{C}$  measurements for the ND-113 tree.

GrN	Rings	$^{14}\text{C}$ (yr BP)	$\sigma$	$\delta^{13}\text{C}$ (‰)	cal BP
26246	9–18	13,375	100	-28.51	16,534
26247	19–28	13,310	75	-27.41	16,524
26248	29–38	13,150	60	-27.22	16,514
26249	39–48	13,215	55	-26.71	16,504
26250	49–58	13,195	55	-26.77	16,494
26251	59–68	13,160	50	-26.98	16,484
26252	69–78	13,365	55	-26.91	16,474
26253	79–88	13,365	55	-25.81	16,464
26254	89–98	13,240	55	-26.11	16,454
26255	99–108	13,335	55	-25.68	16,444
26256	109–118	13,280	55	-26.45	16,434
26257	119–128	13,320	70	-26.65	16,424
26258	129–138	13,265	60	-26.75	16,414
26259	139–148	13,325	60	-26.93	16,404
26260	149–158	13,295	80	-27.15	16,394
26261	159–168	13,240	65	-26.99	16,384
26262	169–178	13,230	60	-27.13	16,374
26263	179–188	13,280	55	-26.43	16,364
26264	189–198	13,260	65	-26.80	16,354
26265	199–208	13,240	55	-26.94	16,344
26266	209–218	13,310	55	-26.91	16,334
26267	219–228	13,220	55	-27.17	16,324
26268	229–238	13,230	55	-27.03	16,314
26269	239–248	13,280	55	-27.16	16,304
26270	249–258	13,245	55	-26.82	16,294
26271	259–268	13,305	35	-26.64	16,284
26272	269–278	13,245	55	-26.20	16,274
26273	279–288	13,220	55	-26.73	16,264
26274	289–298	13,215	55	-26.42	16,254
26275	299–308	13,140	45	-26.86	16,244
26276	309–318	13,185	40	-26.41	16,234
26277	319–328	13,160	55	-26.68	16,224
26278	329–338	13,225	55	-27.01	16,214
26279	339–348	13,105	50	-26.88	16,204

The statistical precision of the individual AMS dates (Fuji tree) is 0.4–0.5%. Because of the multiple analyses performed, this has been reduced to typically 40–60 BP. The precision of the conventional dates (ND-113 tree) ranges from 40 to 100 BP, depending on sample size. There was not enough wood available for high-precision dating. All  $^{14}\text{C}$  determinations have been rounded to the nearest 5.

## DISCUSSION

Pleistocene floating tree-ring sequences of tree rings spanning centuries potentially provide important information on aspects of natural  $^{14}\text{C}$  variations and  $^{14}\text{C}$  timescale calibration. The tree found near Mt. Fuji has 400 rings and has been thoroughly dated by AMS. It is wiggle-matched to the calibration curve IntCal09 (Reimer et al. 2009). The results of the wiggle-match are shown in Figure 3. The overall trend of the Fuji data is consistent with IntCal09. This particular tree dated to  $\sim 23,500$  yr ago. The averaged  $^{14}\text{C}$  dates (Table 1) are plotted individually, the IntCal09 curve with its error envelope is shown as well, as given by the OxCal program.

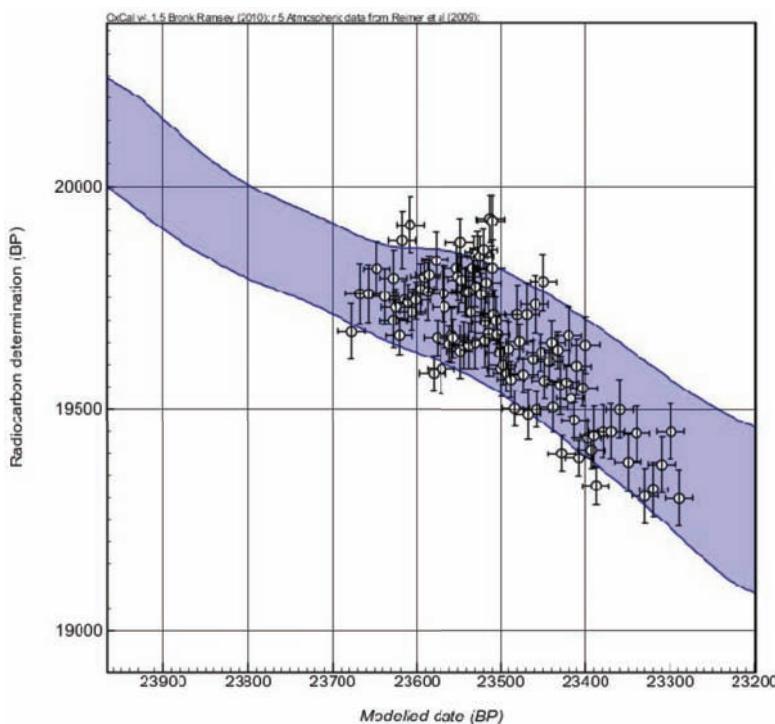


Figure 3 Radiocarbon-dated tree-ring series from the Fuji tree, matched to the IntCal09 calibration curve.

The relevant part of the IntCal curve is derived from marine data. Thus, it is subject to reservoir corrections. Any change in reservoir corrections obviously changes the wiggle-match date of the Fuji tree. More important for the discussion here, IntCal09 is a smoothed curve for the relevant time range, based on a limited amount of data. For the time range of the Fuji tree, the IntCal09 data set shows only 6 data points from the Cariaco data set (Reimer et al. 2009).

The Fuji record is a truly terrestrial one, and is not smoothed but shows significant variations in the atmospheric  $^{14}\text{C}$  content. There appear to be wiggles in the data, but it is not possible to draw conclusions on solar variations because that requires both a better precision and a longer tree sequence. Nevertheless, the data are instrumental for the assessment of calibration curve variability in the IntCal data set (P J Reimer, personal communication).

Tree ND-113 is a floating sequence of ~350 yr, dating to ~16,400 yr ago. The result of the wiggle-matching to IntCal09 is shown in Figure 4. Also, this part of IntCal09, to which our atmospheric data set is matched, is marine derived. The most remarkable feature is the large wiggle at 16,500 cal BP. There are 4 measurements that are consistent; it is not an outlier. At present, there is no explanation for this large wiggle. It is in any case lacking in the marine data set used for IntCal09 (Reimer et al. 2009). In terms of  $\Delta^{14}\text{C}$ , the amplitude of this wiggle is 35‰.

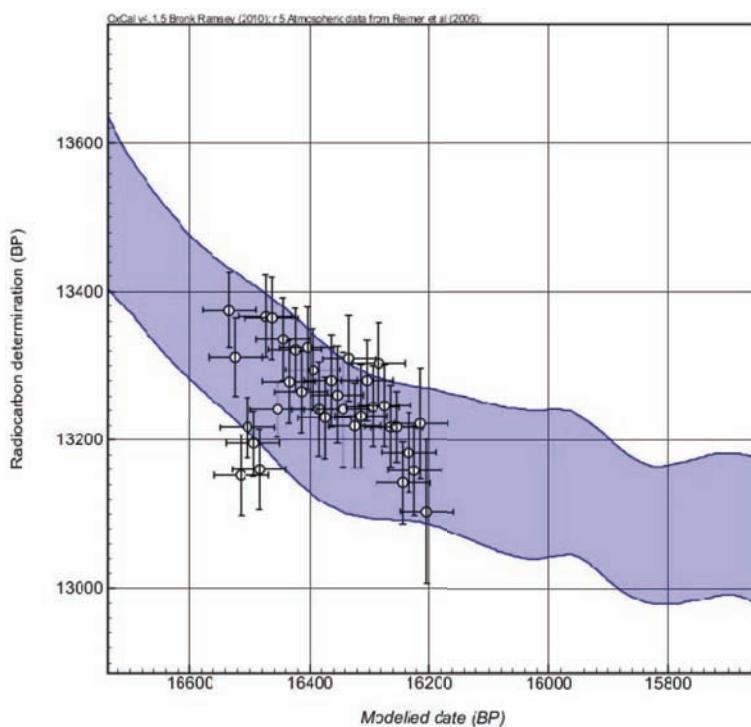


Figure 4 Radiocarbon-dated tree-ring series from ND-113, matched to the IntCal09 calibration curve.

It is interesting to note that a similar excursion in another floating tree-ring series from Japan is observed, albeit at younger age of 15,600 cal BP (Horiuchi et al. 2007). Also, this wiggle is a negative excursion in  $\Delta^{14}\text{C}$  of about 35‰.

Our analysis shows that  $^{14}\text{C}$  dating of floating Pleistocene long-lived tree-ring series contributes to our knowledge of natural  $^{14}\text{C}$  variations and to ongoing calibration work in general. More work is in progress as such trees are found at different locations. Another example from Japan is a tree dating to 22,000 BP, reported by Sato et al. (2010).

Floating tree-ring series are also available from other regions. Of course, there is kauri wood from New Zealand (Balter 2006; Turney et al. 2007). Further, we are presently analyzing the Paleolithic site Kurtak in Siberia (Haesaerts et al. 2005). Trees have been found there that date back to 32,000 BP. However, these trees lived only ~100 yr, which is significantly younger than the trees from Japan presented here.

## CONCLUSION

We have  $^{14}\text{C}$  dated series of tree rings from 2 Late Pleistocene trees from Japan. They show ~350 and ~400 annual rings for the ND-113 tree and Fuji tree, respectively. Both tree-ring sequences date beyond the tree-ring part of the  $^{14}\text{C}$  calibration curve, and are dendrochronologically floating. Tree ND-113 has been dated by the conventional method, while the Fuji tree was dated by AMS.

Both sequences are wiggle-matched to the calibration curve IntCal09. This resulted in an age range of 16,534–16,204 cal BP for ND-113, and 23,678–23,290 cal BP for the Fuji tree. The matching errors are 44 (ND-113) and 16 (Fuji) calendar years. Our results are useful in assessing the variability in the IntCal data set.

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