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HIGH-PRECISION ^{14}C MEASUREMENT OF GERMAN AND IRISH OAKS TO SHOW THE NATURAL ^{14}C VARIATIONS FROM 7890 TO 5000 BC

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INTRODUCTION

The availability of absolutely (dendrochronologically) dated German oak has allowed the Belfast laboratory to extend its published high-precision ^{14}C measurements of Irish oak (Pearson *et al.* 1986) by 2680 yr. The samples were selected at contiguous 20-yr intervals, following a precedent adopted and considered satisfactory in previous publications. All samples were measured for at least 200,000 counts within the ^{14}C channel. The statistical counting error, together with the error on standards, backgrounds and applied corrections, give a realistic precision quoted on each sample of ± 2.5‰ (± 20 yr). This error is considered high-precision for sample ages of 7000–8000 BP.

In May 1986, the Palaeoecology Centre at The Queen's University of Belfast moved into a new, specially designed building with *ca.* 300 m^2 of laboratory space, plus an additional 75 m^2 of underground counting room (overhead shielding of 1.5 m of high-density concrete). The counting room is temperature-controlled to ± 1°C, and is isolated electrically from the main laboratory. Two LKB Wallac Quantulus counters, modified to the authors' specifications, were purchased in 1987, and were used for the high-precision ^{14}C measurements presented in this paper. Details of counter adjustments, methods of quality control and the corrections applied to the ^{14}C measurements will be discussed elsewhere; some relevant details are given below.

We performed duplicate analyses (Table 1) of six samples to confirm internal consistency. In 1986, the University of Arizona Laboratory of Isotope Geochemistry, Tucson, presented high-precision ^{14}C measurements of decadal bristlecone pine samples (Linick *et al.* 1986). Comparison of some of the Arizona measurements, covering a period of 300 yr, to the Belfast data, shows no significant bias between the mean values of the two data sets.

HIGH-PRECISION ^{14}C MEASUREMENT

The techniques used for the combustion of sample carbon and its conversion to benzene have remained essentially the same in our new laboratory, although completely new, redesigned combustion and synthesis lines have been installed. The methods are detailed in previous publications (Pearson 1979, 1980, 1983; Pearson & Baillie 1983; Pearson *et al.* 1986). The fundamental requirements for high-precision ^{14}C measurement do not change; they are to measure only pure uncontaminated sample carbon under known stable and standardized counting conditions. Many variables that had to be resolved by correction in our old laboratory, using a Phillips counter (now > 15 yr old), have been reduced to an insignificant level, using LKB Wallac 'Quantulus' counters. The efficiency of the ^{14}C counting channel is approximately the same as before (*ca.* 70%), but the background for a 15-ml benzene blank has reduced from > 9 cpm to *ca.* 1.2 cpm, using the same glass vials. This benefit has been achieved in proportion by *ca.* 2–3 cpm from the increased overhead shielding, and the remainder from the advantageous design and critical operation of the 'Quantulus' counters.

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The ratio of modern to background count rate is *ca.* 100:1, which, together with long-term stability necessary to give constant counting conditions, now makes high-precision ^{14}C measurement very much easier than with the older system. Detailed analysis of background has shown that the backgrounds used for ^{14}C age calculations could be underestimated by *ca.* 0.10 cpm. This would make all the Belfast dates a few years older at 8000 BC, but only 1 or 2 years different for modern samples. Because this is a variable quantity, and presently not accurately quantifiable (due to contamination of lithium metal used in benzene synthesis), we have not considered it in these age calculations. We re-evaluated one correction (efficiency variation with time) used in the analysis of previously published Belfast data, which significantly changes some dates (average is *ca.* 16 yr older). The published dates presented earlier have been corrected accordingly. A full list of corrected data is also presented (Pearson & Qua 1993).

TABLE 1. Full Replicate Analyses of Calibration Samples

Cal BC*	Date measured	^{14}C age BP**	Published dates – present dates (yr)
3130	Feb/Mar 1980	4506 ± 15	−22
3130	Mar/Apr 1988	4528 ± 20	
3150	Dec 1976/Jan 1977	4553 ± 14	28
3150	Mar/Apr 1988	4525 ± 20	
3170	Feb/Mar 1980	4496 ± 18	21
3170	Mar/Apr 1988	4475 ± 16	
3190	Dec 1976/Jan 1977	4540 ± 15	18
3190	Jan/Feb 1980		
3190	Mar/Apr 1988	4522 ± 20	
3210	Dec 1976/Jan 1977	4560 ± 16	35
3210	Mar/Apr 1988	4525 ± 20	
3230	Jan/Feb 1977	4528 ± 12	2
3230	Oct/Nov 1979		
3230	Mar/Apr 1988	4526 ± 20	
			Mean difference = 13.6

*Cal BC = midpoint of bidecadal sample
**^{14}C age BP = age of bidecadal sample + 1σ

To help justify a claim to accuracy, and at the same time, help to determine a laboratory error multiplier, both replicate analysis and interlaboratory comparisons are necessary. We measured six contiguous samples to give an overall precision of ± 20 yr on each sample. They gave a mean age difference of *ca.* 13 yr, when compared to the same samples (some already duplicated) measured some 8–11 yr before (Table 1). This difference is considered reasonable, although just at the acceptable limit of statistical expectation. We compared recent replicate analysis of Irish oak samples from 5170–5090 BC to German oak, and the mean values differed by <10 yr. A comparison of German oak with previously reported Belfast data, corrected as above, suggested a significant bias between both data sets. However, taking these new measurements into account, we now consider that both data sets are within statistical expectations. This is consistent with the fact that both the German and Irish oak chronologies are absolute.

RADIOCARBON TIME-SCALE CALIBRATION

The longest single unbroken record of high-precision ^{14}C measurements of dendrochronologically-dated wood was published in the special *RADIOCARBON* Calibration Issue (Pearson *et al.* 1986). Some 7000 yr of decadal and bidecadal measurements of Irish oak were presented graphically, and are used as a 'Radiocarbon Time-Scale Calibration'. These curves are now extended by another 2680 yr, forming a complete sequence back to 7980 BC, giving almost 10,000 yr of high-precision time-scale calibration.

At the Twelfth ^{14}C Conference (1985) in Trondheim, it was agreed that definitive calibration would be published by combining data for time periods that had been duplicated independently by different laboratories, provided they showed agreement within statistical expectation. Two papers resulted (Stuiver & Pearson 1986; Pearson & Stuiver 1986), which covered a time period of some 4500 yr between 2500 BC and the present. The mean difference between the Belfast and Seattle data sets was 0.6 yr with a standard deviation of 25.6 yr (Stuiver & Pearson 1986).

When the data set errors were multiplied by their respective error multiplier of 1.23 (Belfast) and 1.6 (Seattle), the resultant standard deviation was found to be 22.9 yr. Thus, the laboratory precision accounted for almost all variability between the data sets.

We compared data presented here with those of Linick *et al.* (1986) covering a 700-yr period. No significant consistent bias is apparent between both data sets, although the agreement is outside of statistical expectation, based on quoted errors. Thus, before these data can be combined, appropriate error multipliers must be determined to provide realistic standard deviations. We hope that this can be accomplished in the near future. The same conclusions are reached when Belfast data are compared with those of the Heidelberg Laboratory (Kromer *et al.* 1986). However, because the Heidelberg samples are not contiguous, and quite often only one annual growth ring has been measured, it is much more difficult to compare realistically both data sets. More work is needed before these data sets can be combined.

Figure 1A–F shows the natural ^{14}C variations over the time period, 7890–5000 BC. Table 2 gives individual ^{14}C ages in yr BP and $\Delta\ ^{14}C$ values, together with respective precisions.

CONCLUSION

We consider the measurements carried out in our new laboratory with LKB Wallac Quantulus counters as accurate as any of our previous measurements. We expect that the quoted precisions underestimate the true standard deviation, because additional errors in background have not been taken into account. We also expect that an error multiplier between 1.2 and 1.4 would be required to provide a realistic estimate of the true standard deviation. If this is so, and if it is taken that the dendrochronology is absolute, then some conclusions drawn by Linick *et al.* (1986), particularly in the suggested positions of maxima and minima, are suspect, and give cause for re-evaluation. However, because the comparison between Linick *et al.* (1986) and the Belfast data gives only a small difference in the mean values, it supports an assumption of accuracy for each laboratory's measurements, albeit with erroneous precision. Further, if the accuracy (*i.e.*, no significant bias) is assumed genuine, and if additional laboratory cross-checks on identical material (to establish meaningful error multipliers) can explain the observed larger-than-statistically-valid differences, then we must conclude that the German oak and bristlecone pine chronologies are in agreement over this period. The agreement between Irish and German oak measurements also supports this conclusion. The six replicated analyses of identical samples in the Belfast lab showed good agreement and consistency in analysis.

There appears to be nothing unusual about this 2680-yr extension to the Radiocarbon Time Scale Calibration. Quite small calendrical band-width increases will be encountered when calibrating conventional ^{14}C ages over much of this 2600-yr extension of the ^{14}C timescale; however, a flattening of the curve between 6700 and 7000 BC presents substantial increases in the calendrical band-width of calibrated dates.

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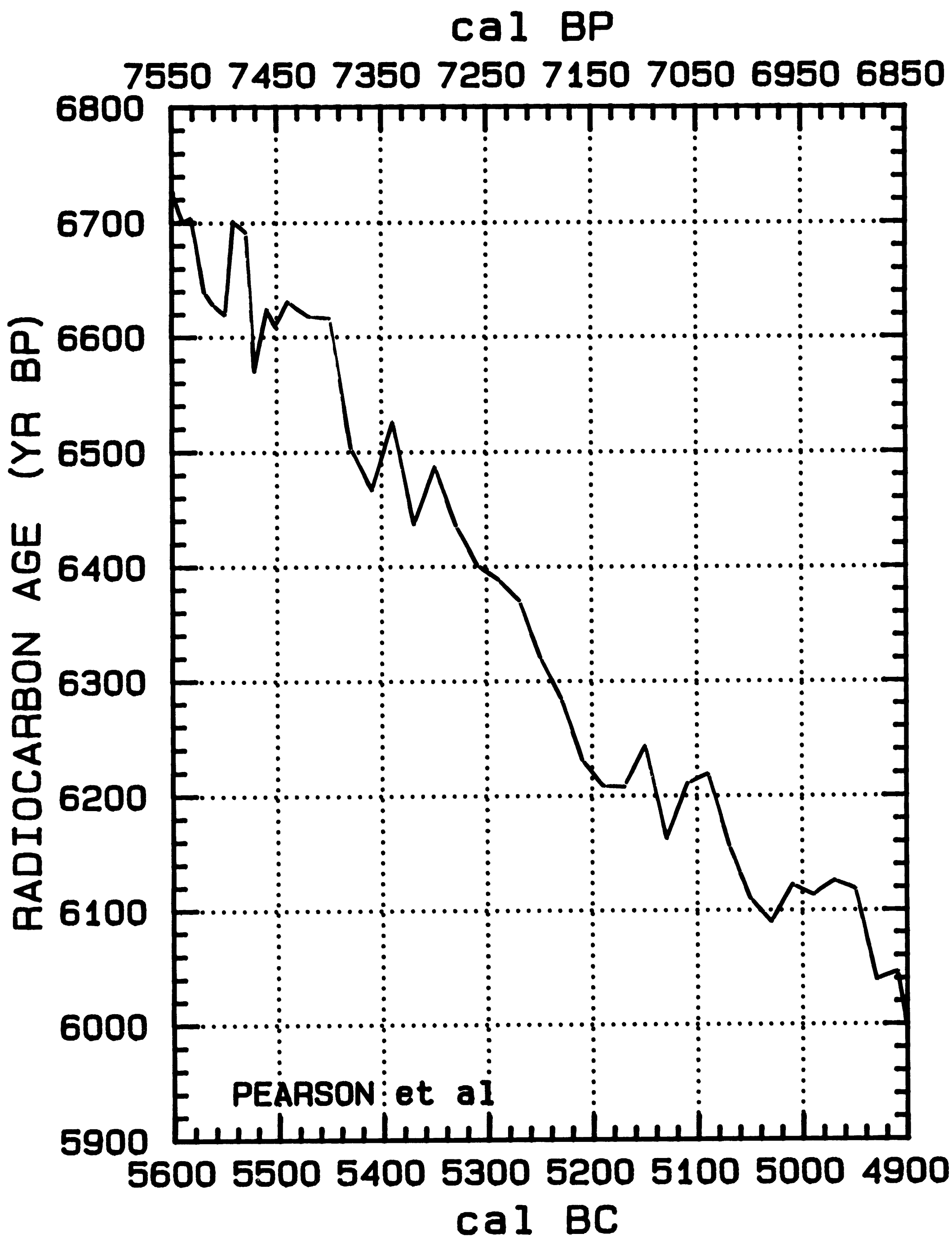

Fig. 1A–F. Calibration curve derived from bidecadal samples

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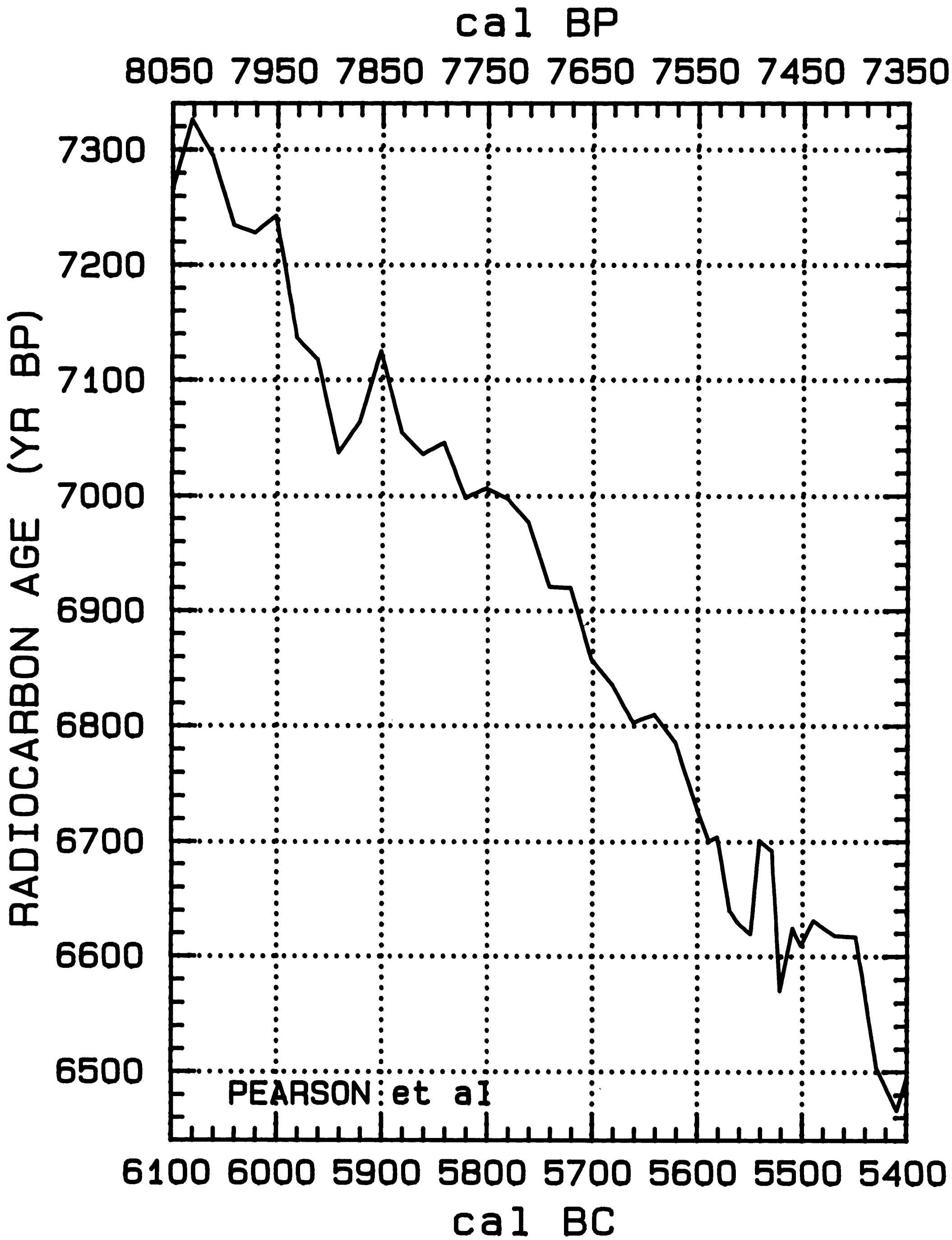

Fig. 1B

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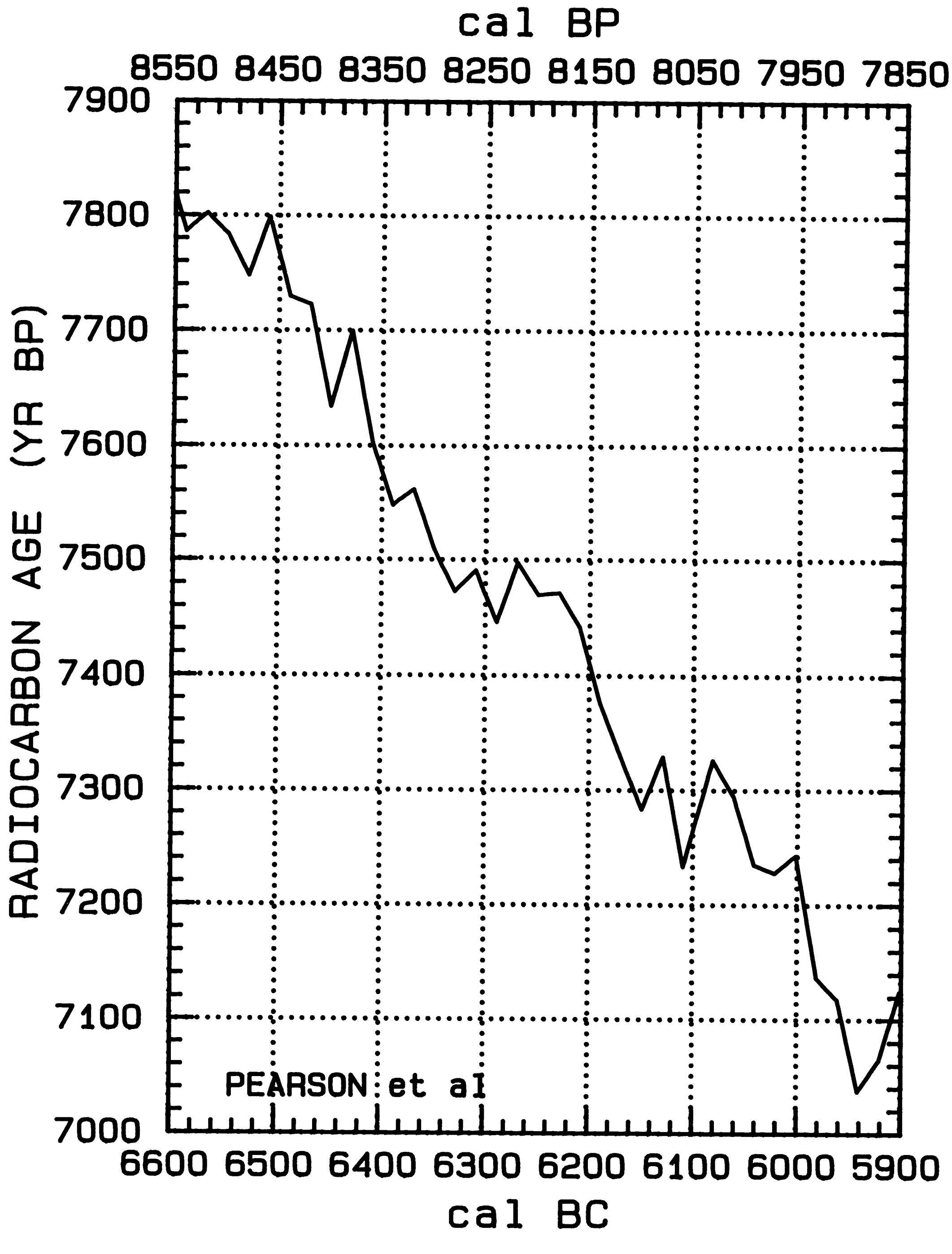

Fig. 1C

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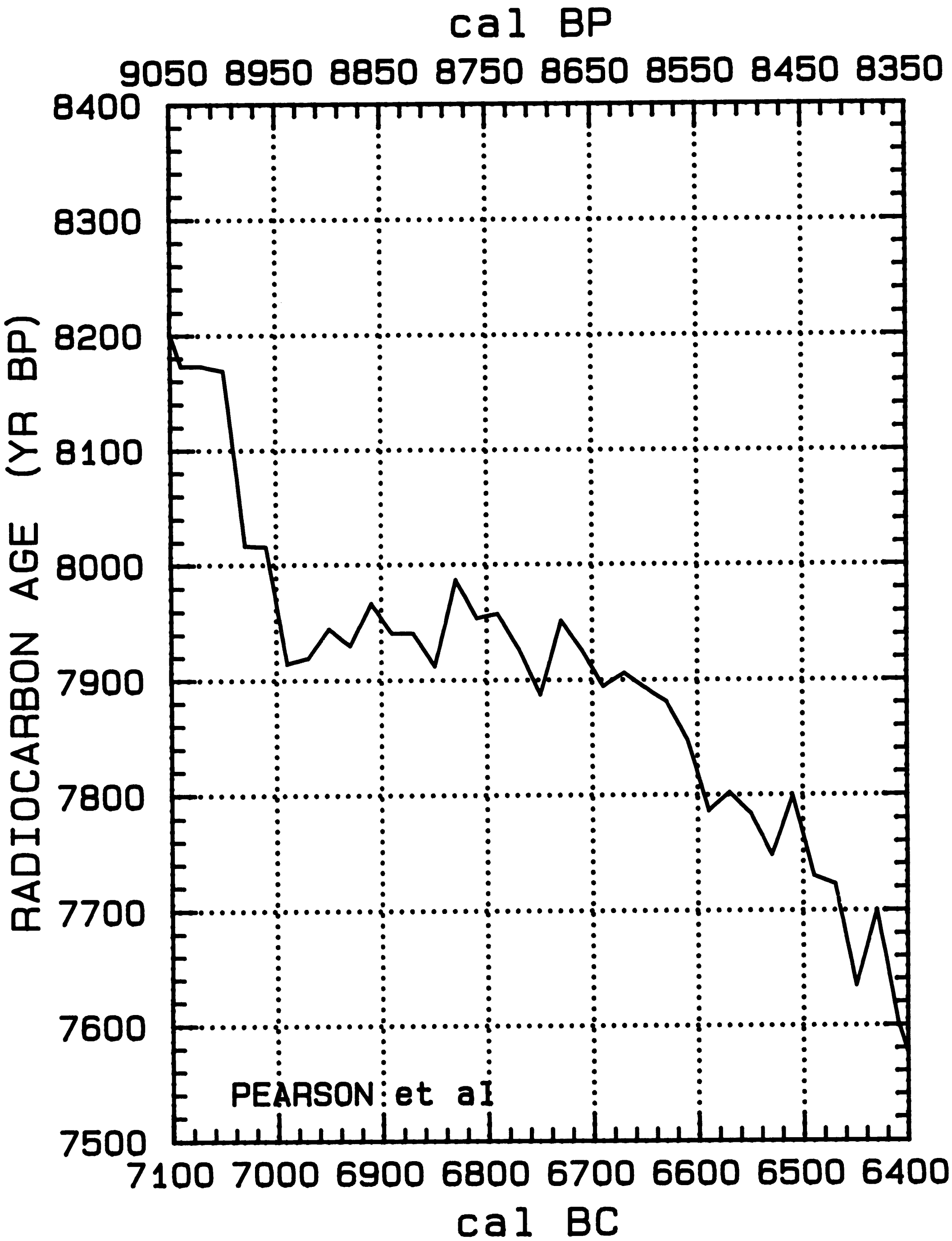

Fig. 1D

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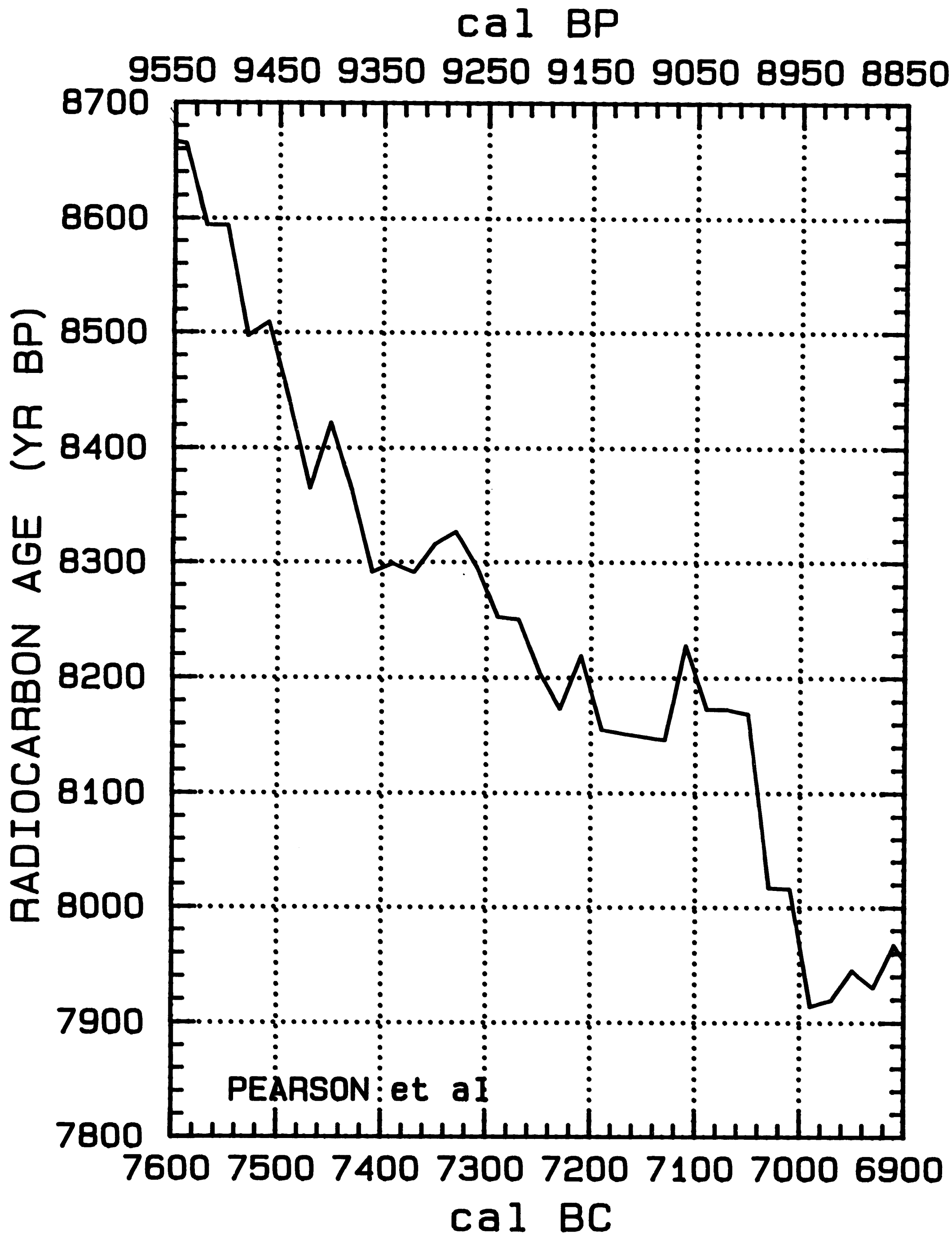

Fig. 1E

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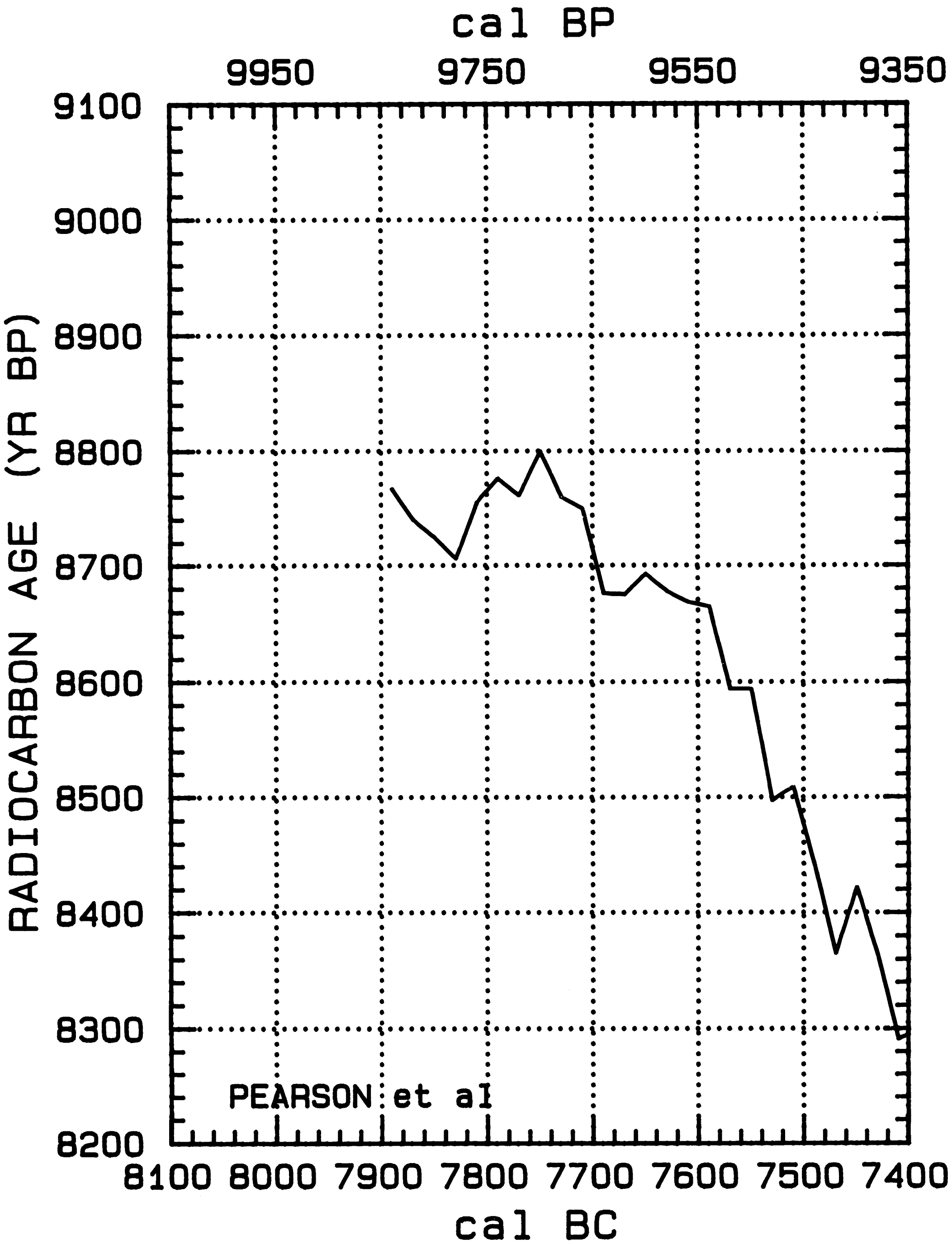

Fig. 1F

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TABLE 2. ^{14}C Ages Over the Time Period, 7890–5000 BC

Cal AD/BC	$\Delta^{14}C$ ‰	^{14}C age (BP)	Cal BP	Cal AD/BC	$\Delta^{14}C$ ‰	^{14}C age (BP)	Cal BP
5010 BC	83.1 ± 2.2	6122 ± 16	BP 6960	5842 BC	67.6 ± 2.4	7046 ± 18	BP 7792
5030 BC	90.2 ± 2.5	6089 ± 18	BP 6980	5862 BC	71.5 ± 2.4	7036 ± 18	BP 7812
5050 BC	89.9 ± 2.4	6110 ± 17	BP 7000	5882 BC	71.6 ± 2.4	7055 ± 18	BP 7832
5070 BC	86.2 ± 1.7	6157 ± 13	BP 7020	5902 BC	64.9 ± 2.4	7125 ± 18	BP 7852
5090 BC	80.5 ± 1.5	6219 ± 11	BP 7040	5922 BC	75.6 ± 2.5	7064 ± 18	BP 7872
5110 BC	84.3 ± 1.5	6210 ± 11	BP 7060	5942 BC	81.8 ± 2.5	7037 ± 18	BP 7892
5130 BC	93.4 ± 1.5	6162 ± 11	BP 7080	5962 BC	73.5 ± 2.5	7118 ± 18	BP 7912
5150 BC	85.1 ± 1.5	6243 ± 11	BP 7100	5982 BC	73.6 ± 2.6	7137 ± 19	BP 7932
5170 BC	92.6 ± 1.5	6207 ± 11	BP 7120	6002 BC	62.1 ± 2.6	7243 ± 19	BP 7952
5190 BC	95.1 ± 2.2	6208 ± 16	BP 7140	6022 BC	66.7 ± 2.6	7228 ± 19	BP 7972
5210 BC	94.6 ± 2.2	6231 ± 16	BP 7160	6042 BC	68.3 ± 2.6	7235 ± 19	BP 7992
5230 BC	89.9 ± 2.2	6285 ± 16	BP 7180	6062 BC	62.9 ± 2.6	7295 ± 19	BP 8012
5250 BC	87.7 ± 2.2	6321 ± 16	BP 7200	6082 BC	61.4 ± 2.6	7326 ± 19	BP 8032
5270 BC	83.6 ± 2.2	6371 ± 16	BP 7220	6102 BC	72.7 ± 2.6	7260 ± 19	BP 8052
5290 BC	83.7 ± 2.2	6389 ± 16	BP 7240	6110 BC	77.4 ± 2.6	7233 ± 19	BP 8060
5310 BC	84.6 ± 2.2	6402 ± 16	BP 7260	6130 BC	67.2 ± 2.6	7329 ± 19	BP 8080
5330 BC	82.7 ± 2.2	6436 ± 16	BP 7280	6150 BC	75.9 ± 2.6	7283 ± 19	BP 8100
5350 BC	78.4 ± 2.2	6487 ± 16	BP 7300	6170 BC	72.5 ± 2.3	7328 ± 17	BP 8120
5370 BC	87.9 ± 2.2	6436 ± 16	BP 7320	6190 BC	68.8 ± 2.3	7375 ± 17	BP 8140
5390 BC	78.4 ± 2.2	6526 ± 16	BP 7340	6210 BC	62.5 ± 2.3	7442 ± 17	BP 8160
5410 BC	89.1 ± 2.2	6466 ± 16	BP 7360	6230 BC	61.1 ± 2.3	7472 ± 17	BP 8180
5430 BC	86.6 ± 2.2	6504 ± 16	BP 7380	6250 BC	63.9 ± 2.3	7470 ± 17	BP 8200
5450 BC	74.0 ± 2.2	6617 ± 16	BP 7400	6270 BC	62.8 ± 2.4	7498 ± 18	BP 8220
5470 BC	76.5 ± 2.2	6618 ± 16	BP 7420	6290 BC	72.3 ± 2.4	7446 ± 18	BP 8240
5490 BC	77.3 ± 2.2	6631 ± 16	BP 7440	6310 BC	68.9 ± 2.4	7491 ± 18	BP 8260
5502 BC	81.9 ± 2.5	6609 ± 18	BP 7452	6330 BC	73.9 ± 2.4	7473 ± 18	BP 8280
5510 BC	80.9 ± 2.2	6624 ± 16	BP 7460	6350 BC	71.7 ± 2.4	7509 ± 18	BP 8300
5522 BC	89.8 ± 2.5	6570 ± 18	BP 7472	6370 BC	67.2 ± 2.4	7562 ± 18	BP 8320
5530 BC	74.4 ± 2.2	6692 ± 16	BP 7480	6390 BC	71.7 ± 2.4	7548 ± 18	BP 8340
5542 BC	74.7 ± 2.4	6701 ± 18	BP 7492	6410 BC	66.9 ± 2.4	7603 ± 18	BP 8360
5550 BC	86.8 ± 2.2	6619 ± 16	BP 7500	6430 BC	56.7 ± 2.4	7700 ± 18	BP 8380
5562 BC	87.0 ± 3.4	6629 ± 25	BP 7512	6450 BC	68.0 ± 2.8	7634 ± 21	BP 8400
5570 BC	86.6 ± 2.2	6640 ± 16	BP 7520	6470 BC	58.8 ± 2.4	7723 ± 18	BP 8420
5582 BC	79.5 ± 2.5	6704 ± 18	BP 7532	6490 BC	60.4 ± 2.4	7730 ± 18	BP 8440
5590 BC	81.1 ± 2.2	6700 ± 16	BP 7540	6510 BC	53.9 ± 2.4	7799 ± 18	BP 8460
5602 BC	78.5 ± 2.5	6731 ± 18	BP 7552	6530 BC	63.2 ± 2.7	7748 ± 18	BP 8480
5622 BC	73.8 ± 2.4	6786 ± 18	BP 7572	6550 BC	61.0 ± 2.7	7784 ± 19	BP 8500
5642 BC	73.1 ± 2.4	6810 ± 18	BP 7592	6570 BC	61.2 ± 2.7	7802 ± 19	BP 8520
5662 BC	76.7 ± 2.5	6803 ± 18	BP 7612	6590 BC	65.8 ± 2.7	7786 ± 20	BP 8540
5682 BC	74.9 ± 2.4	6836 ± 18	BP 7632	6610 BC	60.2 ± 2.6	7848 ± 19	BP 8560
5702 BC	74.5 ± 2.4	6858 ± 18	BP 7652	6630 BC	58.4 ± 2.6	7881 ± 19	BP 8580
5722 BC	68.8 ± 2.4	6920 ± 18	BP 7672	6650 BC	59.4 ± 2.2	7893 ± 16	BP 8600
5742 BC	71.3 ± 2.4	6921 ± 18	BP 7692	6670 BC	60.3 ± 2.2	7906 ± 16	BP 8620
5762 BC	66.4 ± 2.4	6977 ± 18	BP 7712	6690 BC	64.4 ± 2.2	7894 ± 16	BP 8640
5782 BC	66.2 ± 2.4	6998 ± 18	BP 7732	6710 BC	62.7 ± 2.2	7926 ± 16	BP 8660
5802 BC	67.6 ± 2.4	7007 ± 18	BP 7752	6730 BC	61.9 ± 2.2	7952 ± 16	BP 8680
5822 BC	71.4 ± 2.4	6998 ± 18	BP 7772	6750 BC	73.1 ± 2.6	7887 ± 19	BP 8700

TABLE 2. (Continued)

Cal AD/BC	Δ14C ‰	14C age (BP)	Cal BP	Cal AD/BC	Δ14C ‰	14C age (BP)	Cal BP
6770 BC	70.4 ± 2.6	7927 ± 19	BP 8720	7370 BC	99.9 ± 2.5	8291 ± 18	BP 9320
6790 BC	68.8 ± 2.6	7958 ± 19	BP 8740	7390 BC	101.5 ± 2.8	8299 ± 20	BP 9340
6810 BC	71.9 ± 2.6	7954 ± 19	BP 8760	7410 BC	105.3 ± 2.4	8291 ± 17	BP 9360
6830 BC	70.1 ± 2.6	7987 ± 19	BP 8780	7430 BC	97.9 ± 2.4	8364 ± 17	BP 9380
6850 BC	82.8 ± 1.7	7912 ± 12	BP 8800	7450 BC	92.7 ± 2.4	8422 ± 17	BP 9400
6870 BC	81.5 ± 2.7	7941 ± 20	BP 8820	7470 BC	103.1 ± 2.4	8365 ± 17	BP 9420
6890 BC	84.1 ± 2.7	7941 ± 20	BP 8840	7490 BC	95.4 ± 2.4	8441 ± 17	BP 9440
6910 BC	83.2 ± 2.7	7967 ± 20	BP 8860	7510 BC	88.8 ± 2.5	8509 ± 18	BP 9460
6930 BC	90.9 ± 2.8	7930 ± 20	BP 8880	7530 BC	93.0 ± 2.5	8497 ± 18	BP 9480
6950 BC	91.5 ± 2.8	7945 ± 20	BP 8900	7550 BC	82.5 ± 3.4	8594 ± 25	BP 9500
6970 BC	97.7 ± 2.8	7919 ± 20	BP 8920	7570 BC	85.2 ± 2.5	8594 ± 18	BP 9520
6990 BC	101.0 ± 2.8	7914 ± 20	BP 8940	7590 BC	78.2 ± 2.5	8665 ± 18	BP 9540
7010 BC	89.7 ± 2.8	8016 ± 20	BP 8960	7610 BC	80.3 ± 2.5	8669 ± 18	BP 9560
7030 BC	92.2 ± 2.8	8017 ± 20	BP 8980	7630 BC	81.7 ± 2.5	8678 ± 18	BP 9580
7050 BC	74.4 ± 1.7	8169 ± 12	BP 9000	7650 BC	82.3 ± 2.5	8693 ± 18	BP 9600
7070 BC	76.4 ± 2.0	8173 ± 15	BP 9020	7670 BC	87.3 ± 2.5	8675 ± 18	BP 9620
7090 BC	79.0 ± 2.1	8173 ± 15	BP 9040	7690 BC	89.9 ± 2.5	8676 ± 18	BP 9640
7110 BC	74.3 ± 2.0	8228 ± 15	BP 9060	7710 BC	82.5 ± 2.7	8750 ± 20	BP 9660
7130 BC	87.9 ± 2.6	8146 ± 19	BP 9080	7730 BC	83.7 ± 2.7	8760 ± 20	BP 9680
7190 BC	94.6 ± 2.6	8155 ± 19	BP 9140	7750 BC	81.0 ± 2.7	8800 ± 20	BP 9700
7210 BC	88.6 ± 2.6	8219 ± 19	BP 9160	7770 BC	88.9 ± 2.8	8761 ± 20	BP 9720
7230 BC	97.5 ± 2.6	8173 ± 19	BP 9180	7790 BC	89.5 ± 2.8	8776 ± 20	BP 9740
7250 BC	95.8 ± 2.5	8205 ± 18	BP 9200	7810 BC	95.0 ± 2.5	8755 ± 18	BP 9760
7270 BC	92.1 ± 2.5	8251 ± 18	BP 9220	7830 BC	104.3 ± 2.5	8706 ± 18	BP 9780
7290 BC	94.5 ± 2.4	8253 ± 17	BP 9240	7850 BC	104.4 ± 2.5	8725 ± 18	BP 9800
7310 BC	91.3 ± 2.5	8296 ± 18	BP 9260	7870 BC	105.0 ± 2.5	8740 ± 18	BP 9820
7330 BC	89.7 ± 2.8	8327 ± 20	BP 9280	7890 BC	104.0 ± 2.5	8767 ± 18	BP 9840
7350 BC	93.9 ± 2.8	8316 ± 20	BP 9300				