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**Characterisation, Optimisation, and Standard Measurements for
Two Small-Sample High-Precision Radiocarbon Counters**

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Characterisation, Optimisation, and Standard Measurements for Two Small-Sample High-Precision Radiocarbon Counters

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Summary

In February 1998 English Heritage provided a grant for two small-sample high-precision counters to extend the capacity for this sort of radiocarbon measurement at the Queen's University, Belfast Radiocarbon Laboratory. This report explains the processes used for liquid scintillation counter characterisation, optimisation, and benzene purity correction and provides details of background, oxalic acid standard, and known-age wood measurements. The counters are now operational.

Keywords

Radiocarbon

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CHARACTERISATION, OPTIMISATION, AND STANDARD MEASUREMENTS FOR TWO SMALL-SAMPLE HIGH-PRECISION RADIOCARBON COUNTERS

Introduction

At the end of 1996, a new system for small-sample high-precision radiocarbon dating became available to commercial collaborators at the Queen's University, Belfast Radiocarbon dating Laboratory. This followed the successful completion of an NERC Research Grant (GR/H26055).

In order to expand the capacity for this new dating method to meet the demand of projects funded by English Heritage, an infrastructure grant for two new liquid scintillation counters was agreed.

This report describes the processes and measurements carried out to commission this equipment for small-sample high-precision radiocarbon measurement.

Installation

Two Quantulus 1220 liquid scintillation counters were purchased from EG&G Wallac Ltd who delivered, installed, and commissioned them between 13 May 1998 and 20 May 1998. Both counters were modified as requested by the Belfast Radiocarbon Laboratory, to have manual high voltage adjustment in line with all the existing Quantulus counters in the laboratory. Both counters have been installed in a purpose built, shielded basement laboratory in the Palaeoecology Centre at Queen's University, Belfast. They are now designated as Q5 and Q6.

Vial characterisation and background measurements

In setting up a counter for high precision measurement all possible sources of variability of the ^{14}C count rate need to be minimised. For this reason 200 low-potassium glass vials were weighed, and a catalogue of vial weights was produced (see Appendix 1). One of the set was broken and discarded. Each vial was examined for optical quality and uniformity of glass and a further 23 vials were discarded. Of the remaining 176 vials, two sets of 10 with the smallest weight range were selected as counting vials, ie the variance in total glass weight for the vial set was minimised (see Appendix 2). Chrome-plated brass caps were selected to fit these vials. The sealing and vial filling technique are as described in Pearson (1983).

Each of the 10 vials for each counter was filled with 2.6265g of analar benzene (ie no ^{14}C) and 0.05g of butyl PBD. The background activity of the vials was measured across a range of channel intervals (Tables 1 and 2). The average counts per minute for all 20 vials were determined and the six closest to the mean in each counter were selected for use. This is a lengthy process and was begun before the counters were optimised. Background counts per

minute (CPM) are shown for channels 200-600 and the final counting window used, ie, Q5 channel 108-568 and Q6 channel 104-568. Tables 1 and 2 shows the vials selected as the initial set (others are retained and used when breakage occurs). In addition, weight loss due to evaporation over the four months counting time was determined (Tables 3a-b show weight loss data for each vial). The selected vials fell within accepted limits for weight loss (< 0.0003 g per day). On only one occasion was weight loss actually used as a selection parameter.

Oxalic acid II standards

Twelve samples of oxalic acid II (NBS 19) were converted to carbon dioxide by wet oxidation and synthesised to benzene according to methods outlined in Wilson *et al* (1996). Each was counted in one of the selected vials. The average counts per minute per gram of benzene, after background subtraction, $\delta^{13}\text{C}$ fractionation correction, correction for ^{14}C contamination during synthesis, and correction to the AD 1890 atmospheric ^{14}C level (Stuiver and Polach 1977), are given in Tables 4 and 5. These samples provide an indication of the vial to vial variability for samples standards because the benzene was synthesised as a homogenised batch. The six oxalic acid samples for each counter, each measured in a different vial, verify the reproducibility from a single known-activity standard. In addition they, along with four additional oxalics prepared independently for each counter, provide a basis for future oxalic acid determinations for dating samples of unknown age.

Counter characterisation and optimisation

Sets of high voltage measurements were made on the counters when the automatic high voltage option was used. The results are given in Appendix 3. The measurements were so stable that the possibility of automatic control of high voltage was considered. The requirement of small-sample high-precision measurement, to keep overall counting times as small as possible and precision as high as possible, placed a premium on maximising count rate. For this reason a series of tests was run to determine if open window counting and automatic high voltage control could be used. An example of the results of the tests are given in Appendix 4. Calculations were made based on the balance point window described in McCormac (1992) and using a fully open window. The effect on date determination was shown to be negligible. This experiment confirmed the viability of automatic control and open window configuration. These settings have been used for configuration of the instruments.

Precise determination of error multipliers requires a considerable quantity of data. Preliminary analysis suggested that the multiplier for the system lay between 1 and 1.25. A conservative value of 1.25 was used in initial calculations. Further measurements (Tables 6a-b which give error multipliers of 1.21 and 1.04 for Q5 and Q6 respectively) have shown that this is indeed conservative. However, because of problems encountered with Q5 we have continued to use 1.25 and all results given in this report use this value.

Dendrochronology

Sixteen 100g samples of oak (*Quercus patrea* and *Quercus rubra*) from the decades centered at AD 725 to AD 575 were supplied to the Belfast Radiocarbon Laboratory on the 18 - 19 January 1999. The samples were mainly extracted from one wood sample Q3693. Four radiocarbon samples centering on AD 605, AD 595, AD 585, and AD 575 had some wood from sample Q3685. The wood was obtained from structural timbers of a horizontal mill from the townland of Brabstown, Co Kilkenny, Ireland. The location of this site is at latitude 52° 40' N and longitude 7° 24' W. Sample Q3693 is from the cross-piece of the mill while sample Q3685 is from a collapsed upright. These tree-ring series from these two samples are a part of the Brabstown master chronology which is 333 years in length and dates from AD 549 to AD 881. This chronology gives extremely significant consistent correlation values with a series of local and national chronologies.

Dendrochronological methodology

Tree-ring dating for this site was carried out using the methodology described by Baillie (1982). The samples have been prepared by polishing the surface with various grades of emery paper. The tree-ring pattern is then highlighted with teaching chalk. This is rubbed into the polished surface of the sample. The spring and summer pores retain the chalk and provide better visual definition of the ring widths under the microscope.

Measurement of the tree-ring pattern was carried out using a Henson measuring stage. This machine measures the annual ring width in 1/50mm. The tree-ring series obtained from the samples was plotted on graph paper for visual comparison by a dendrochronologist. The correlation values in Table 7 were obtained by using the tree-ring cross-dating program called Cross84 (Munro 1984). Extremely significant and consistent correlation values were obtained for samples Q3693 and Q3685.

Brabstown tree-ring chronology

A mean master chronology was formed using the samples listed in Table 7. This chronology is 333 years in length. It was then compared with a series of site chronologies from the southern part of Ireland. Extremely significant and consistent correlation values were found. These results are listed in Table 8. These results indicate that the Brabstown chronology dates from AD 549 to AD 881. A comparison was made with a tree-ring chronology called the North Mills Index Master (Nthmills). This is an independent chronology from the northern part of Ireland. The Brabstown chronology gave an extremely significant and consistent correlation value ($t = 5.72^{***}$) with this chronology.

Known-age wood standards

Each decadal sample was pre-treated to alpha cellulose (Hoper *et al* 1998). The samples were then divided into two parts and combusted, synthesised to benzene, and counted independently. The results are given in Table 9 and are plotted with respect to Pearson *et al*

(1986) in Figure 1, Pearson *et al* (1986) (decadal replicates combined and averaged) in Figure 2, INTCAL98 Stuiver *et al* (1998a) in Figure 3, and The University of Washington decadal measurements on north American conifers Stuiver *et al* (1998b) in Figure 4. Table 10 gives the offsets of the measurements we have made on decadal oak with respect to these datasets. The offset with respect to the Pearson *et al* (1986) calibration data set is -3.8 ± 7.6 yrs. With respect to INTCAL98 the offset is -16.1 ± 6.2 radiocarbon years, and between the replicates of decadal oak measured in the series it is 1.4 ± 8.7 yrs.

Conclusions

The two counters funded by English Heritage have been installed and commissioned at the Queen's University, Belfast Radiocarbon Laboratory. Sets of vials have been prepared, and standard and background data accumulated. Replicate pairs of decadal samples of oak (Figures 1 – 4) show the excellent agreement of measurements made on the system with previous calibration data and highlight the need to create bespoke segments of calibration for the resolution of chronological problems in difficult time periods such as that of Anglo-Saxon England.

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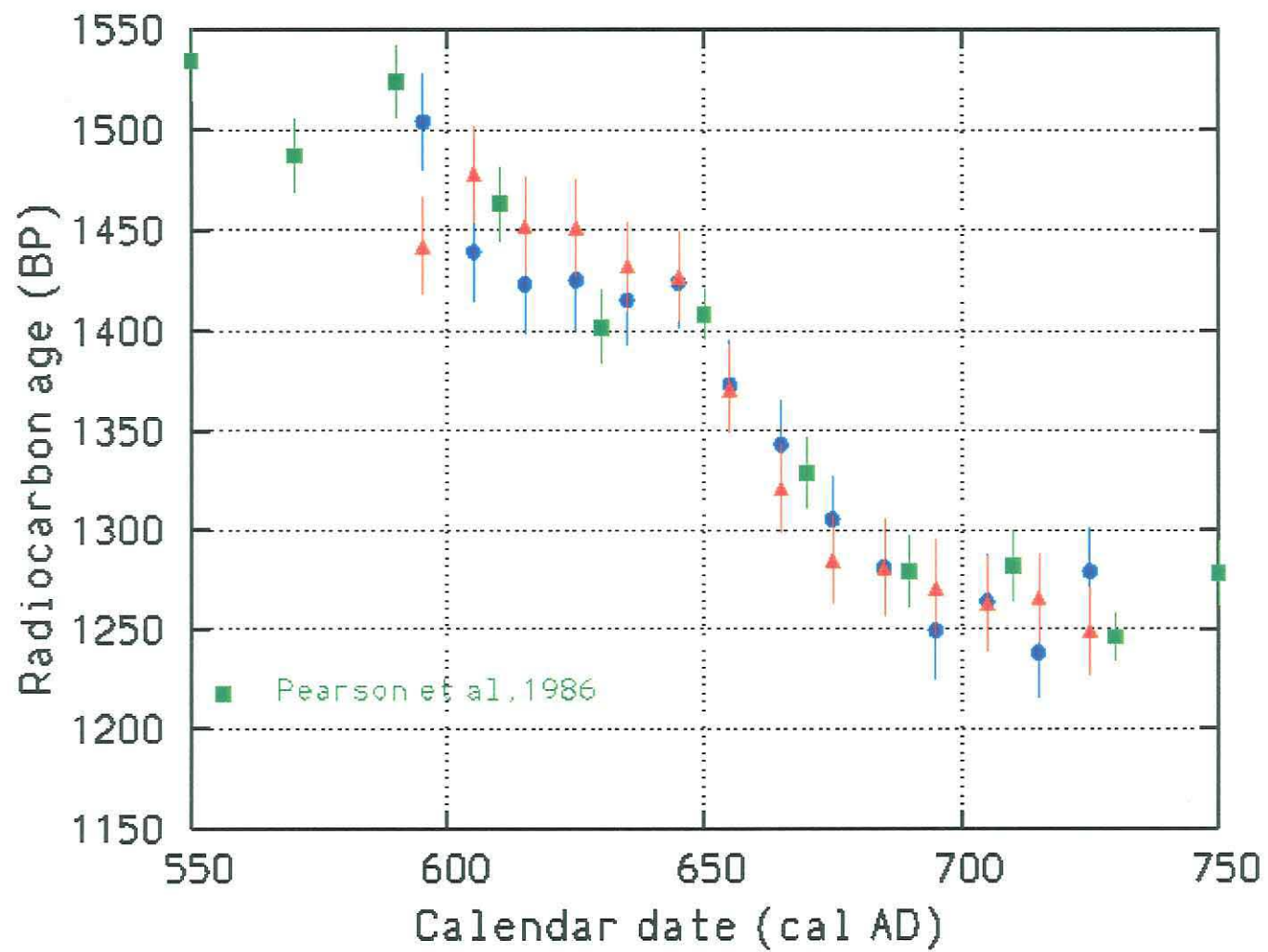


Figure 1. Brabstown decadal replicates plotted with respect to Pearson *et al* (1986)

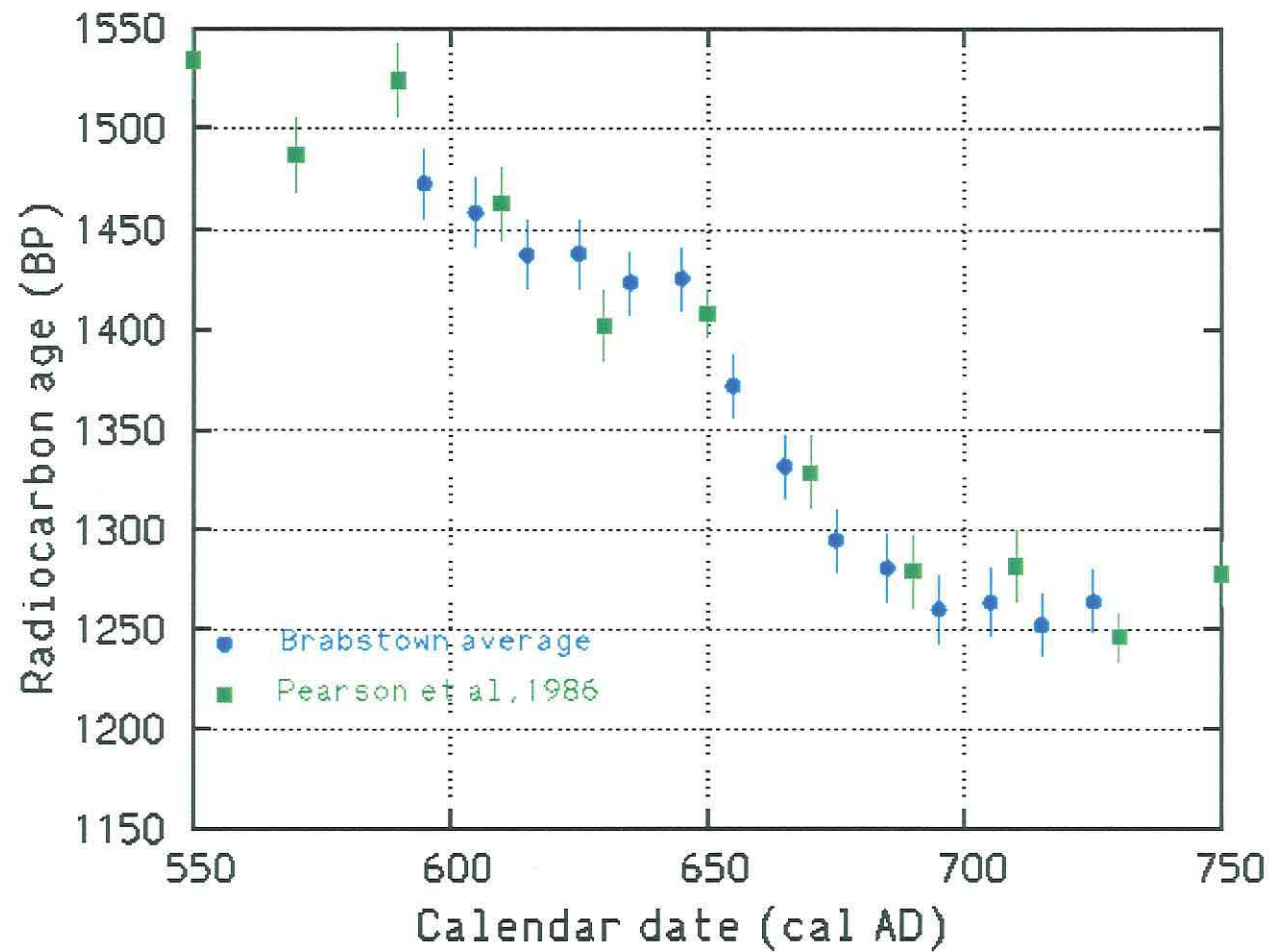


Figure 2. Weighted averages of Brabstown decadal replicates plotted with respect to Pearson *et al* (1986)

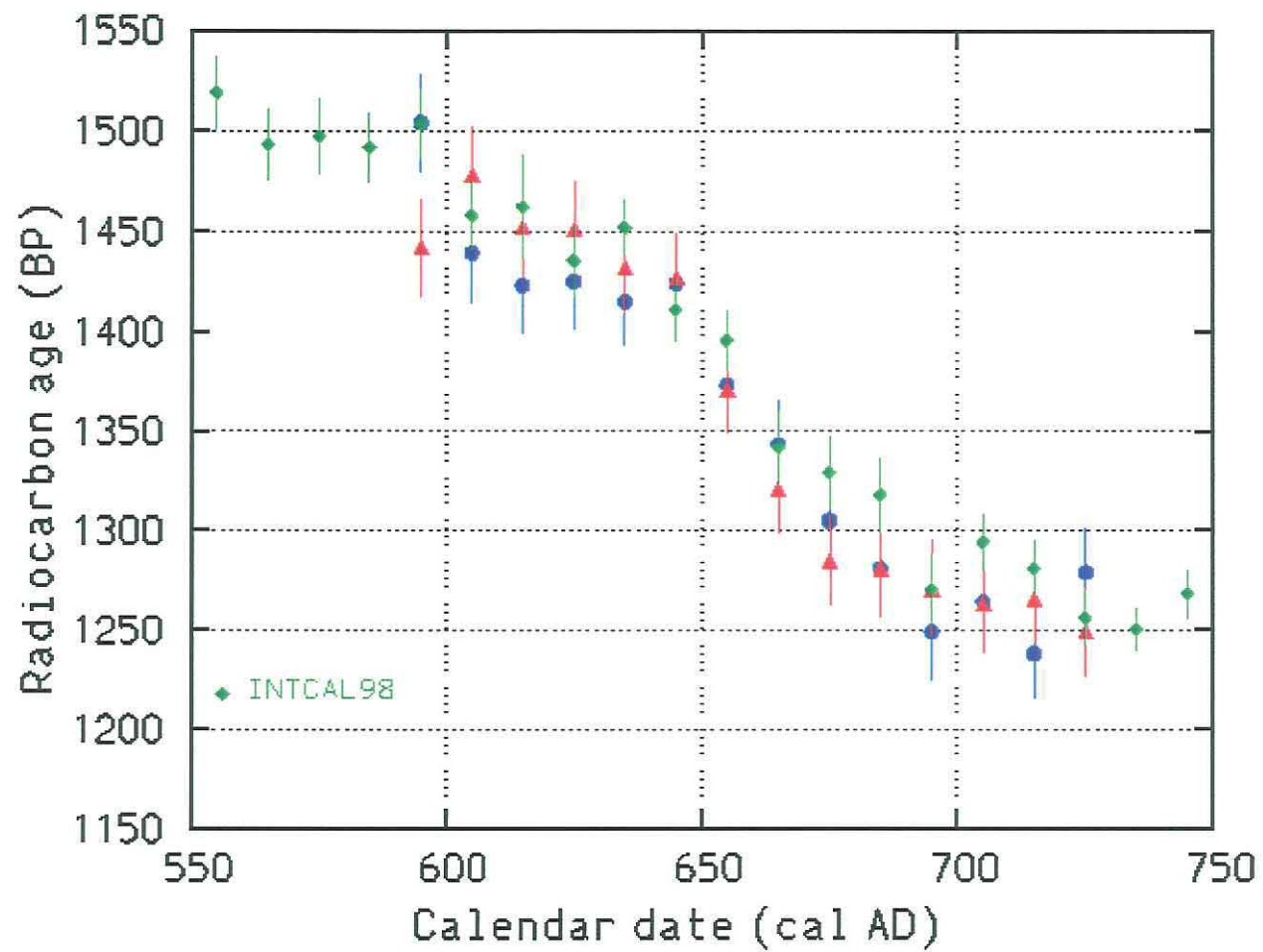


Figure 3. Brabstown decadal replicates plotted with respect to INTCAL98 (Stuiver *et al* 1998a)

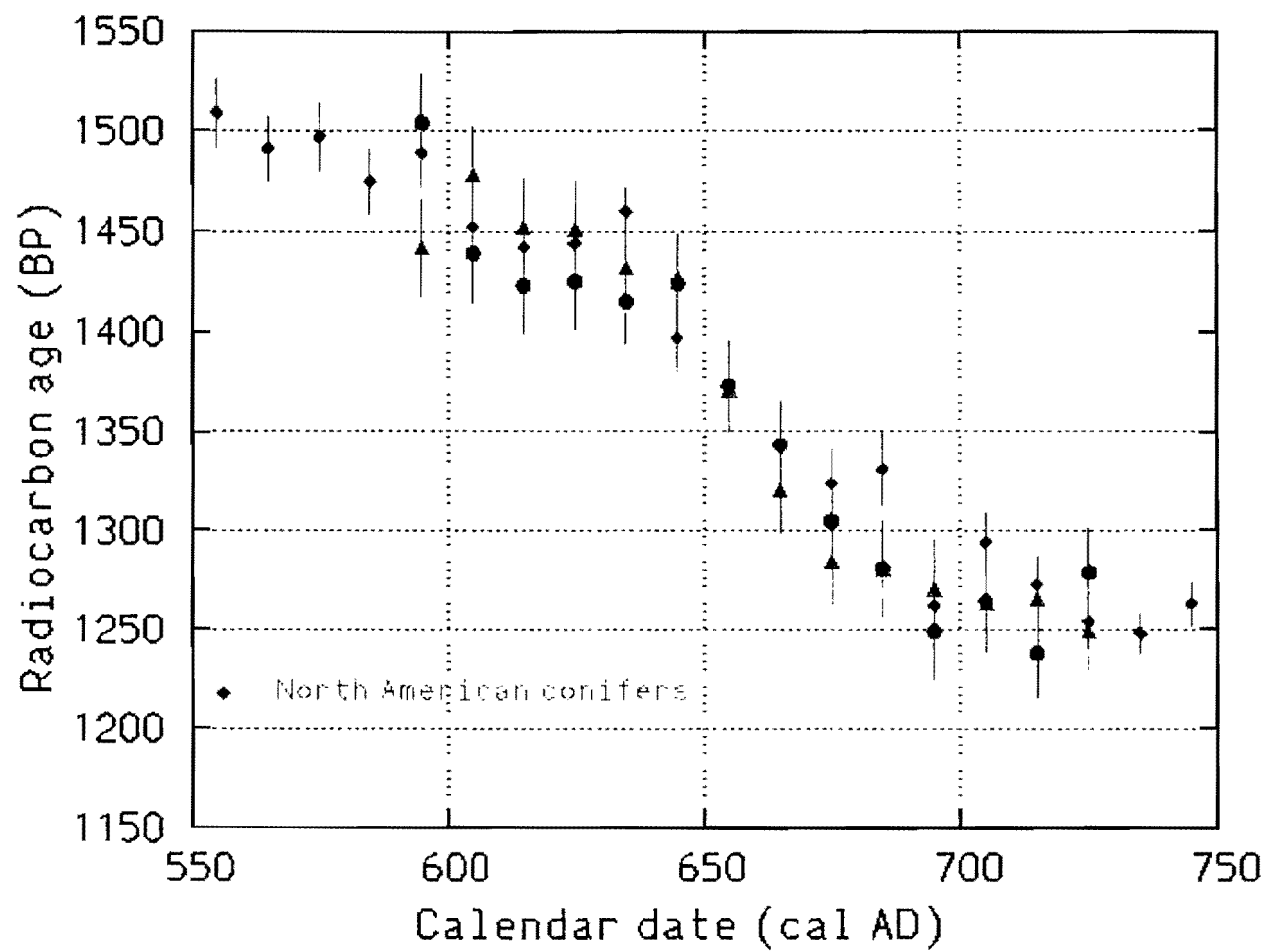
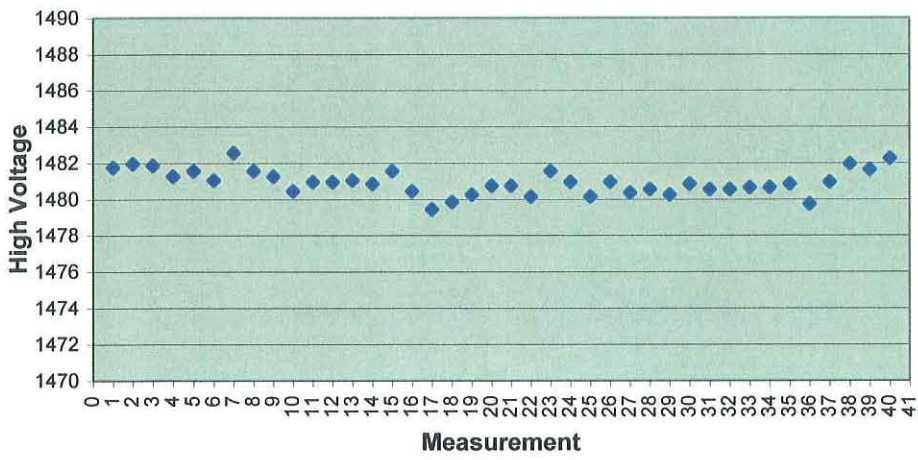
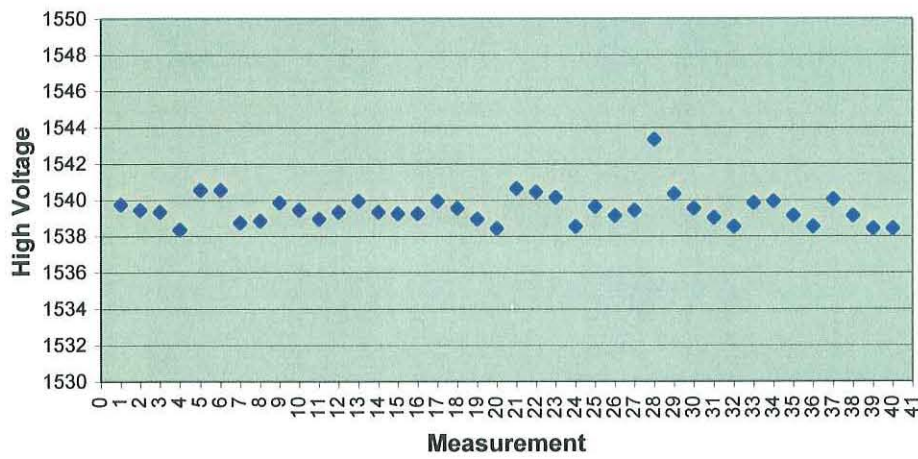


Figure 4. Brabstown decadal replicates plotted with respect to north American conifers (Stuiver *et al* 1998b)

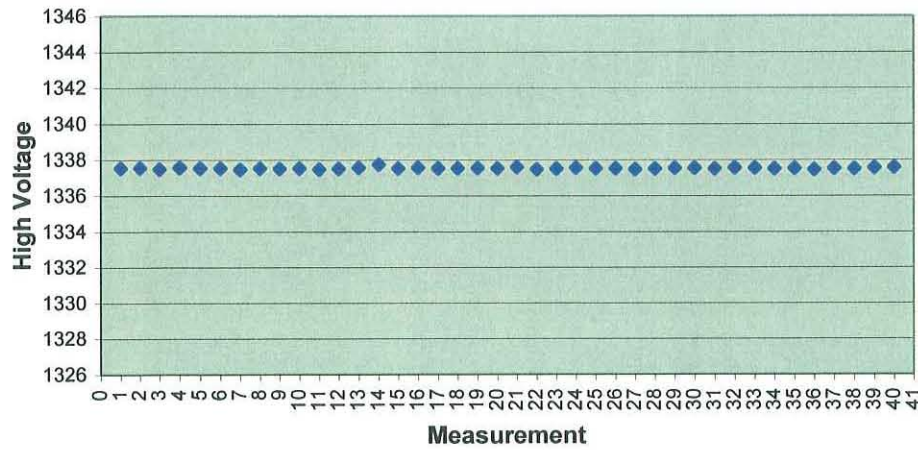
Q5 Left PMT High Voltage Measurements



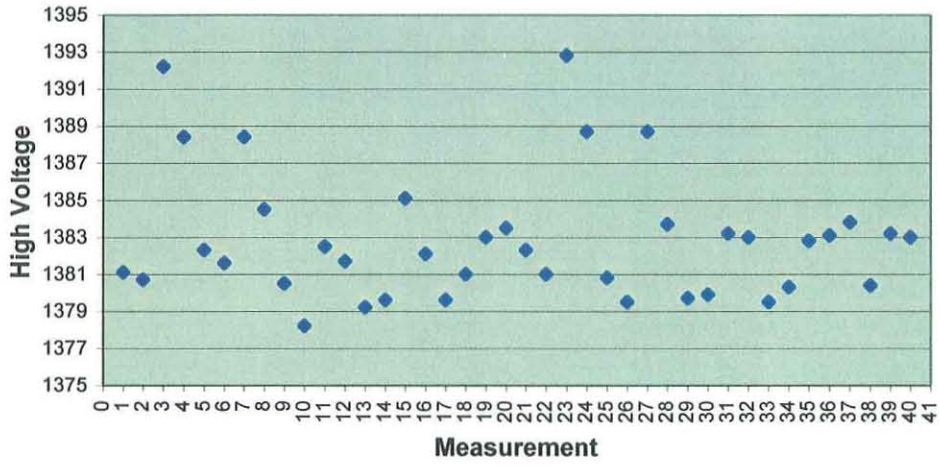
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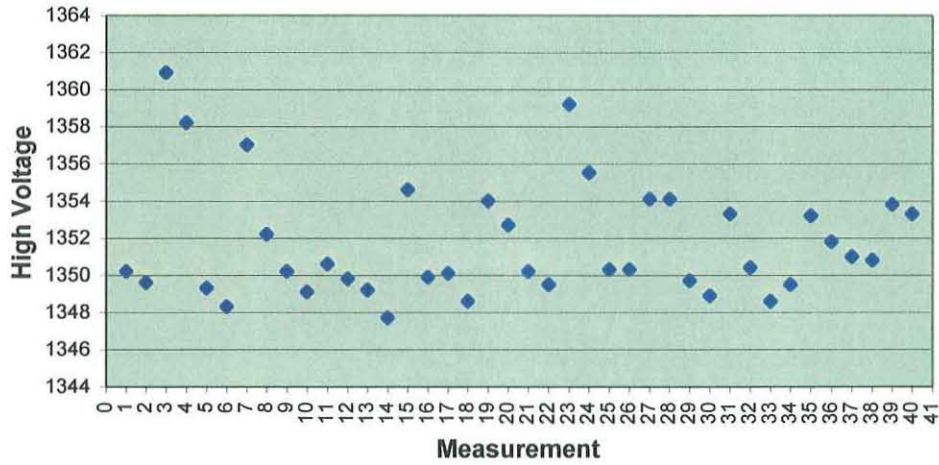
Q5 Guard High Voltage Measurements



Q6 Left PMT High Voltage Measurements



Q6 Right PMT High Voltage Measurements



Q6 Guard High Voltage Measurements

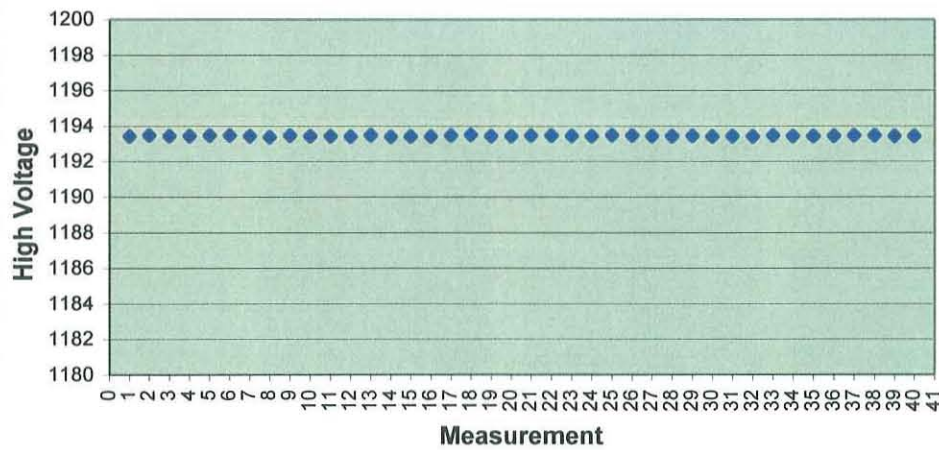


Table 1. Q5 vial selection according to deviation from mean counts per minute (CPM) in a background (BGD) count

Vial	Mean BGD CPM (04/06/98 - 05/10/98) Ch 200-600	Deviation from mean	Order of dev. from mean	Mean BGD CPM (04/06/98 - 05/10/98) Ch 108-568	Deviation from mean	Order of dev. from mean
16	0.7160	-0.0080	5	0.7820	-0.0039	1
33	0.7000	0.0080	6	0.7690	0.0091	5
46	0.7360	-0.0280	9	0.8250	-0.0469	10
55	0.6670	0.0410	10	0.7430	0.0351	9
73	0.7100	-0.0020	1	0.7740	0.0041	2
111	0.7240	-0.0160	8	0.7900	-0.0119	7
118	0.7040	0.0040	2	0.7710	0.0071	3
138	0.7150	-0.0070	3	0.7870	-0.0089	4
144	0.6930	0.0150	7	0.7500	0.0281	8
172	0.7150	-0.0070	4	0.7900	-0.0119	6
Mean=	0.7080			0.7781		

Table 2. Q6 vial selection according to deviation from mean counts per minute (background count)

Vial	Mean BGD CPM (04/06/98 - 05/10/98) Ch 200-600	Deviation from mean	Order of dev. from mean	Mean BGD CPM (04/06/98 - 05/10/98) Ch 108-568	Deviation from mean	Order of dev. from mean
1	0.7630	0.0046	3	0.8760	-0.0031	1
2	0.7720	-0.0044	2	0.8800	-0.0071	4
19	0.7740	-0.0064	4	0.8790	-0.0061	3
28	0.7530	0.0146	8	0.8400	0.0329	9
82	0.7600	0.0076	5	0.8550	0.0179	7
120	0.7560	0.0116	6	0.8650	0.0079	5
140	0.7910	-0.0234	10	0.9180	-0.0451	10
145	0.7560	0.0116	7	0.8500	0.0229	8
164	0.7680	-0.0004	1	0.8770	-0.0041	2
195	0.7830	-0.0154	9	0.8890	-0.0161	6
Mean=	0.7676			0.8729		

6 vials with smallest deviation from mean CPM

Note: Weight loss considered to decide between selection of vial 82 (0.00474g) and vial 195 (0.00665g) above. Vial 82 was selected.

Table 4. Calculation of Q5 corrected Oxalic Acid counts per minute per gram (CPM/g)

Vial Number	Start Weight	Mean Counting Weight	Counts per Minute (CPM)	Mass Spec. Value ($\delta^{13}\text{C}$ ‰)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Syn.fac.	CPM/g	CPM/g corr. for Mass. Spec.	CPM/g corr. for 1950 Value
16	2.62852	2.62539	38.107	-17.952	0.985904	0.782	37.30178	14.20809	14.00781374	10.44842827
33	2.62621	2.624255	38.035	-17.952	0.985904	0.769	37.24278	14.19175	13.99170651	10.43641389
73	2.62712	2.624765	38.165	-17.952	0.985904	0.774	37.36778	14.23662	14.03593989	10.46940757
118	2.6266	2.62247	38.041	-17.952	0.985904	0.771	37.24678	14.20294	14.00273383	10.44463916
138	2.62685	2.621155	37.994	-17.952	0.985904	0.787	37.18378	14.18603	13.98606242	10.43220396
172	2.62779	2.62089	38.134	-17.952	0.985904	0.79	37.32078	14.23974	14.03901205	10.47169909

Table 5. Calculation of Q6 corrected Oxalic Acid counts per minute per gram (CPM/g)

Vial Number	Start Weight	Mean Counting Weight	CPM	Mass Spec. Value ($\delta^{13}\text{C}$ ‰)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Syn.fac.	CPM/g	CPM/g corr. for Mass. Spec.	CPM/g corr. for 1950 Value
1	2.62666	2.623215	37.865	-17.927	0.985854	0.876	36.96578	14.09178	13.89244194	10.36237244
2	2.62709	2.625575	37.798	-17.927	0.985854	0.88	36.89478	14.05208	13.85329554	10.33317314
19	2.62556	2.624265	37.81	-17.927	0.985854	0.879	36.90778	14.06404	13.86509462	10.34197408
82	2.62728	2.625815	37.832	-17.927	0.985854	0.855	36.95378	14.07326	13.87418071	10.34875139
120	2.62641	2.6254	37.745	-17.927	0.985854	0.865	36.85678	14.03854	13.83994972	10.32321849
164	2.62607	2.623655	37.754	-17.927	0.985854	0.877	36.85378	14.04673	13.84802744	10.32924367

Appendix 2. Vial selection according to weight range

	VIAL No.	WEIGHT (g)	Wt.Range of 10 vials (g)	VIAL No.	WEIGHT (g)	Wt.Range of 10 vials (g)	VIAL No.	WEIGHT (g)	Wt.Range of 10 vials (g)	VIAL No.	WEIGHT (g)	Wt.Range of 10 vials (g)
1	108	11.98774	0.10032	51	34	12.16935	101	18	12.23159	151	182	12.31099
2	21	12.01488	0.07403	52	117	12.16953	102	74	12.23307	152	103	12.311
3	15	12.01811	0.07184	53	22	12.17202	103	89	12.23461	153	154	12.312
4	38	12.0199	0.07067	54	3	12.18094	104	61	12.23489	154	194	12.31421
5	30	12.0664	0.02478	55	110	12.1866	105	141	12.23878	155	131	12.31772
6	104	12.08239	0.01141	56	51	12.18769	106	126	12.24052	156	184	12.31939
7	23	12.08374	0.01028	57	127	12.18785	107	68	12.24436	157	85	12.32248
8	27	12.08533	0.01469	58	106	12.19149	108	142	12.24587	158	166	12.32334
9	9	12.08598	0.01582	59	87	12.19183	109	191	12.24641	159	102	12.32513
10	121	12.08806	0.01666	60	4	12.19311	110	136	12.24644	160	193	12.3261
11	6	12.08891	0.01679	61	14	12.19393	111	63	12.25261	161	97	12.32742
12	24	12.08995	0.0161	62	13	12.19535	112	161	12.25313	162	147	12.32905
13	93	12.09057	0.01703	63	107	12.19609	113	52	12.25404	163	153	12.33011
14	48	12.09118	0.02049	64	56	12.19805	114	100	12.25417	164	150	12.33081
15	91	12.0938	0.02077	65	145	12.199	115	37	12.25465	165	71	12.33942
16	20	12.09402	0.0225	66	140	12.1997	116	98	12.2558	166	151	12.34268
17	133	12.10002	0.02123	67	1	12.19977	117	96	12.2567	167	59	12.3456
18	31	12.1018	0.02338	68	82	12.19995	118	109	12.25833	168	90	12.34621
19	58	12.10472	0.02158	69	120	12.20005	119	155	12.26223	169	146	12.35113
20	26	12.1057	0.02281	70	2	12.20055	120	53	12.26355	170	115	12.36179
21	44	12.10605	0.02268	71	28	12.20097	121	183	12.26532	171	137	12.36437
22	78	12.1076	0.0222	72	195	12.20166	122	54	12.26585	172	143	12.36616
23	42	12.11167	0.01925	73	15	12.20322	123	135	12.26656	173	139	12.36629
24	49	12.11457	0.01808	74	163	12.20413	124	124	12.26662	174	114	12.36831
25	35	12.11652	0.01614	75	128	12.20494	125	41	12.268	175	62	12.37101
26	50	12.12125	0.01196	76	55	12.20546	126	152	12.26908	176	181	12.40196
27	99	12.12518	0.01126	77	33	12.20627	127	12	12.26971			
28	72	12.1263	0.01287	78	16	12.20675	128	176	12.27027			
29	84	12.12851	0.01339	79	73	12.2076	129	10	12.27219			
30	148	12.12873	0.01526	80	172	12.20793	130	112	12.27233			
31	40	12.1298	0.01652	81	118	12.20816	131	81	12.27273			
32	43	12.13092	0.02036	82	138	12.20854	132	174	12.27273			
33	57	12.13265	0.01922	83	46	12.21009	133	149	12.27416			
34	11	12.13266	0.02302	84	111	12.21032	134	122	12.27493			
35	8	12.13321	0.02322	85	144	12.2104	135	32	12.2752			
36	77	12.13644	0.02186	86	47	12.21173	136	165	12.27582			
37	130	12.13917	0.02439	87	171	12.21275	137	7	12.27665			
38	70	12.1419	0.0234	88	65	12.21397	138	134	12.27908			
39	29	12.14399	0.02176	89	119	12.21452	139	60	12.27965			
40	80	12.14632	0.01966	90	156	12.21736	140	113	12.28593			
41	94	12.15128	0.01777	91	129	12.21791	141	95	12.28921			
42	76	12.15187	0.01748	92	45	12.21819	142	162	12.29392			
43	36	12.15568	0.01385	93	123	12.2208	143	79	12.29474			
44	5	12.15643	0.01559	94	101	12.22286	144	25	12.29527			
45	66	12.1583	0.02264	95	17	12.22462	145	69	12.29721			
46	185	12.16356	0.02304	96	192	12.22492	146	116	12.29832			
47	86	12.1653	0.02239	97	132	12.22856	147	75	12.30311			
48	92	12.16575	0.0221	98	64	12.23048	148	125	12.30514			
49	83	12.16598	0.02551	99	105	12.23138	149	88	12.30791			
50	39	12.16905	0.02278	100	173	12.23138	150	67	12.3099			

lowest weight range

10 selected vials

Table 3(a). Q5 Weight loss data

Q5 Background Count. Vial weight losses with time.

Vial	Date Filled/start	Weight Loss by 06/07/98	Weight Loss by 03/08/98	Weight Loss by 07/09/98	Weight Loss by 05/10/98
16	04/06/98	0.00223	0.00497	0.00927	0.01493
33	04/06/98	0.00367	0.00664	0.00942	0.01463
46	04/06/98	0.00000	0.00029	0.00154	0.00418
55	04/06/98	0.00128	0.00365	0.00735	0.01159
73	04/06/98	0.00313	0.00682	0.01175	0.01768
111	04/06/98	0.00103	0.00239	0.00559	0.01175
118	04/06/98	0.00101	0.00175	0.00349	0.00633
138	04/06/98	0.00067	0.00125	0.00232	0.00505
144	04/06/98	0.00081	0.00195	0.00476	0.00890
172	04/06/98	0.00019	0.00029	0.00109	0.00365

Table 3(b). Q6 Weight loss data

Q6 Background Count. Vial weight losses with time.

Vial	Date Filled/start	Weight Loss by 03/07/98	Weight Loss by 03/08/98	Weight Loss by 07/09/98	Weight Loss by 05/10/98
1	04/06/98	0.00113	0.00273	0.00500	0.00817
2	04/06/98	0.00087	0.00124	0.00214	0.00464
19	04/06/98	0.00055	0.00067	0.00132	0.00319
28	04/06/98	0.00064	0.00109	0.00233	0.00518
82	04/06/98	0.00011	0.00092	0.00226	0.00474
120	04/06/98	0.00041	0.00046	0.00105	0.00324
140	04/06/98	0.00049	0.00056	0.00123	0.00295
145	04/06/98	0.00014	0.00000	0.00043	0.00258
164	04/06/98	0.00049	0.00065	0.00147	0.00329
195	04/06/98	0.00142	0.00237	0.00387	0.00665

Table 4. Calculation of Q5 corrected Oxalic Acid counts per minute per gram (CPM/g)

Vial Number	Start Weight	Mean Counting Weight	Counts per Minute (CPM)	Mass Spec. Value ($\delta^{13}\text{C} \text{‰}$)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Syn.fac.	CPM/g	CPM/g corr. for Mass. Spec.	CPM/g corr. for 1950 Value
16	2.62852	2.62539	38.107	-17.952	0.985904	0.782	37.30178	14.20809	14.00781374	10.44842827
33	2.62621	2.624255	38.035	-17.952	0.985904	0.769	37.24278	14.19175	13.99170651	10.43641389
73	2.62712	2.624765	38.165	-17.952	0.985904	0.774	37.36778	14.23662	14.03593989	10.46940757
118	2.6266	2.62247	38.041	-17.952	0.985904	0.771	37.24678	14.20294	14.00273383	10.44463916
138	2.62685	2.621155	37.994	-17.952	0.985904	0.787	37.18378	14.18603	13.98606242	10.43220396
172	2.62779	2.62089	38.134	-17.952	0.985904	0.79	37.32078	14.23974	14.03901205	10.47169909

Table 5. Calculation of Q6 corrected Oxalic Acid counts per minute per gram (CPM/g)

Vial Number	Start Weight	Mean Counting Weight	CPM	Mass Spec. Value ($\delta^{13}\text{C} \text{‰}$)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Syn.fac.	CPM/g	CPM/g corr. for Mass. Spec.	CPM/g corr. for 1950 Value
1	2.62666	2.623215	37.865	-17.927	0.985854	0.876	36.96578	14.09178	13.89244194	10.36237244
2	2.62709	2.625575	37.798	-17.927	0.985854	0.88	36.89478	14.05208	13.85329554	10.33317314
19	2.62556	2.624265	37.81	-17.927	0.985854	0.879	36.90778	14.06404	13.86509462	10.34197408
82	2.62728	2.625815	37.832	-17.927	0.985854	0.855	36.95378	14.07326	13.87418071	10.34875139
120	2.62641	2.6254	37.745	-17.927	0.985854	0.865	36.85678	14.03854	13.83994972	10.32321849
164	2.62607	2.623655	37.754	-17.927	0.985854	0.877	36.85378	14.04673	13.84802744	10.32924367

Table 6a. Calculation of error multiplier for Q5

VIAL	OX CPM/G	% OX ERROR	BGD ERROR	ERROR OXALIC	BGD + OXALIC ERROR	ERROR ²
16	10.44840	0.129336	0.002746826	0.013513543	0.013789884	0.000190161
33	10.43640	0.129459	0.002727816	0.013510859	0.013783479	0.000189984
73	10.46940	0.128827	0.002743006	0.013487414	0.013763518	0.000189434
118	10.44460	0.129043	0.002732287	0.013478025	0.013752184	0.000189123
138	10.43220	0.129123	0.002759824	0.013470370	0.013750181	0.000189067
172	10.47170	0.128886	0.002765579	0.013496555	0.013776989	0.000189805

STD DEV 0.01661057

MEAN 10.45045

Error Multiplier 1.206341031

Table 6b. Calculation of error multiplier for Q6

VIAL	OX CPM/G	% OX ERROR	BGD ERROR	ERROR OXALIC	BGD + OXALIC ERROR	ERROR ²
1	10.36237	0.127712	0.003381607	0.013233993	0.013659203	0.000186574
2	10.33320	0.127826	0.003596360	0.013208516	0.013689365	0.000187399
19	10.34200	0.127805	0.003620134	0.013217593	0.013704384	0.000187810
82	10.34880	0.128555	0.003584311	0.013303900	0.013778281	0.000189841
120	10.32320	0.128703	0.003602028	0.013286268	0.013765883	0.000189500
164	10.32920	0.127900	0.003626220	0.013211047	0.013699680	0.000187681

STD DEV 0.01433049

MEAN 10.33980

Error Multiplier 1.044786082

Table 7. Student 't' values for samples, Q3693 and Q3865 cross matching into the Brabstown site chronology.

QUB number	Q3691	Q3693	Q3697	Q3865	Q4748	Q4749
Q3691	-	5.77***	10.91***	4.43**	6.42***	5.04***
Q3693	-	-	5.19**	4.61**	7.12**	3.12vwm
Q3697	-	-	-	3.32nsm	6.09***	-
Q3865	-	-	-	-	3.80*	5.55***
Q4748	-	-	-	-	-	5.72***

Table 8. Student 't' values for the Brabstown site chronology compared with a series of dated local site chronologies.

Site chronology name	Brabstown, Co. Kilkenny
Ballyroe, Co. Wexford	8.54***
Ballyrafton, Co. Kilkenny	7.44***
Newtown, Co. Tipperary	7.18***
Ballygeardra, Co. Kilkenny	6.29***
Ballygormill South, Co. Laois	5.98***
Ballydowane West, Co. Waterford	5.75***
Ballynoe, Co. Cork	5.68***
Farranmareen, Co. Cork	5.47***
Cappagh South, Co. Clare	5.36***
Cloongowna, Co. Clare	5.19***
Templenacroha, Co. Wexford	5.03***

Table 9. Dating of the Brabstown known-age wood series using weighted mean values of all Oxalic II Acid data

Laboratory ID	Sample Decade Centred on	Counter	Sample Corrected CPM/g	Oxalic Weighted 1950 Value	Sample CPM/g corr. for Ox. 1950	¹⁴ C years BP	¹⁴ C years Error	Mass Spec. Value (δ ¹³ C ‰)	Mass Spec. Error
UB-4383	AD 595 S1	Q6	8.54916	10.308899	0.8292990	1504	24	-24.582	0.028
UB-4384	AD 595 S2	Q6	8.61442	10.308899	0.8356295	1442	24	-24.612	0.014
UB-4385	AD 605 S1	Q6	8.61843	10.308899	0.8360185	1439	24	-23.980	0.002
UB-4386	AD 605 S2	Q6	8.57622	10.308899	0.8319240	1478	24	-24.124	0.004
UB-4387	AD 615 S1	Q5	8.73918	10.433449	0.8376118	1423	24	-23.812	0.012
UB-4388	AD 615 S2	Q5	8.70800	10.433449	0.8346233	1452	24	-23.848	0.012
UB-4389	AD 625 S1	Q5	8.73765	10.433449	0.8374652	1425	24	-24.306	0.012
UB-4390	AD 625 S2	Q5	8.70872	10.433449	0.8346923	1451	24	-24.215	0.018
UB-4391	AD 635 S1	Q6	8.64430	10.308899	0.8385280	1415	22	-24.343	0.014
UB-4392	AD 635 S2	Q6	8.62592	10.308899	0.8367450	1432	22	-24.338	0.014
UB-4393	AD 645 S1	Q6	8.63460	10.308899	0.8375870	1424	22	-24.715	0.008
UB-4394	AD 645 S2	Q6	8.63310	10.308899	0.8374415	1425	22	-24.709	0.004
UB-4395	AD 655 S1	Q5	8.79472	10.433449	0.8429351	1373	22	-24.984	0.004
UB-4396	AD 655 S2	Q5	8.79633	10.433449	0.8430894	1371	22	-25.091	0.012
UB-4397	AD 665 S1	Q6	8.72160	10.308899	0.8460263	1343	22	-23.012	0.006
UB-4398	AD 665 S2	Q6	8.74571	10.308899	0.8483651	1321	22	-25.418	0.006
UB-4399	AD 675 S1	Q5	8.86852	10.433449	0.8500085	1305	22	-25.982	0.012
UB-4400	AD 675 S2	Q5	8.89077	10.433449	0.8521410	1285	22	-25.957	0.008
UB-4401	AD 685 S1	Q5	8.89527	10.433449	0.8525723	1281	24	-25.556	0.006
UB-4402	AD 685 S2	Q5	8.89573	10.433449	0.8526164	1281	24	-25.503	0.008
UB-4403	AD 695 S1	Q5	8.93079	10.433449	0.8559768	1249	24	-25.726	0.014
UB-4404	AD 695 S2	Q5	8.90619	10.433449	0.8536190	1271	24	-25.814	0.006
UB-4405	AD 705 S1	Q5	8.91408	10.433449	0.8543752	1264	24	-25.733	0.004
UB-4406	AD 705 S2	Q5	8.91585	10.433449	0.8545448	1263	24	-25.903	0.008
UB-4407	AD 715 S1	Q6	8.83646	10.308899	0.8571682	1238	22	-25.229	0.006
UB-4408	AD 715 S2	Q6	8.80608	10.308899	0.8542212	1266	22	-25.208	0.016
UB-4409	AD 725 S1	Q6	8.79139	10.308899	0.8527962	1279	22	-25.457	0.018
UB-4410	AD 725 S2	Q6	8.82455	10.308899	0.8560128	1249	22	-25.457	0.018

Table 10. Offsets of Brabstown decadal oak measurements

a) sample1 with respect to sample 2

Sample decade centred on	Difference (sample 1-sample2)	Standard deviation
AD 595	62	33.9
AD 605	-39	33.9
AD 615	-29	33.9
AD 625	-26	33.9
AD 635	-17	31.1
AD 645	-3	31.1
AD 655	2	31.1
AD 665	22	31.1
AD 675	20	31.1
AD 685	0	33.9
AD 695	-22	33.9
AD 705	1	33.9
AD 715	-28	31.1
AD 725	30	31.1

Number of comparisons= 14

Average difference= -1.929

Average standard deviation in difference= 32.527

Mean difference= -1.439 +/- 8.669

Std. dev.=sqrt(sample variance)= 27.467

k = Std.dev./Avg. s.d.= 0.844

b) Brabstown bi-decadal average (final) with respect to Belfast data (Pearson *et al* 1986)

Sample bi-decade centred on	Difference (Brabs.ave-Belfast 1986)	Standard deviation
AD 610	-15	21.6
AD 630	28.1	21.4
AD 650	-9.2	16.3
AD 670	-15.5	21.1
AD 690	-8.5	21.6
AD 710	-24.7	21.4
AD 730	18	19.7

Number of comparisons= 7

Average difference=-3.829

Average standard deviation in difference= 20.435

Mean difference= -3.815 +/- 7.612

Std. dev.=sqrt(sample variance)= 18.652

k = Std.dev./Avg. s.d.= 0.913

c) Brabstown decadal replicates with respect to INTCAL98 (Stuiver *et al* 1998a)

Sample decade centred on	Difference (Brabs.reps-INTCAL98)	Standard deviation
AD 595	-30.4	24.8
AD 605	0.7	23.9
AD 615	-24.8	30.7
AD 625	2.8	24.4
AD 635	-28.6	20.8
AD 645	14.4	22.1
AD 655	-23.6	21.5
AD 665	-9.7	24
AD 675	-34.3	24
AD 685	-36.7	25.3
AD 695	-10.5	24.7
AD 705	-30.7	22.2
AD 715	-29.1	20.7
AD 725	7.9	20.6

Number of comparisons= 14

Average difference= -16.614

Average standard deviation in difference= 23.540

Mean difference= -16.120 +/- 6.195

Std. dev.=sqrt(sample variance)= 17.302

k = Std.dev./Avg. s.d.= 0.735

d) Brabstown replicates weighted average with respect to North American conifers (Stuiver *et al* 1998b)

Sample decade centred on	Difference (Brabs.reps-UW conifers)	Standard deviation
595 AD	-16	24
605 AD	6.5	22.7
615 AD	-4.5	36.2
625 AD	-6	23.3
635 AD	-36.5	19.7
645 AD	28.5	23.1
655 AD	1	22.3
665 AD	-10	23.1
675 AD	-29	23.1
685 AD	-50	24.8
695 AD	-2	23.3
705 AD	-30.5	22.7
715 AD	-21	20.3
725 AD	10	20.3

Number of comparisons= 14

Average difference= -11.393

Average standard deviation in difference= 23.495

Mean difference= -11.687 +/- 6.114

Std. dev.=sqrt(sample variance)= 21.030

k = Std.dev./Avg. s.d.= 0.895

Appendix 1. Catalogue of vial weight (g) arranged in ascending order

	VIAL No.	WEIGHT (g)		VIAL No.	WEIGHT (g)		VIAL No.	WEIGHT (g)		VIAL No.	WEIGHT (g)
1	108	11.98774	51	34	12.16935	101	64	12.23048	151	162	12.29392
2	21	12.01488	52	117	12.16953	102	105	12.23138	152	79	12.29474
3	15	12.01811	53	22	12.17202	103	173	12.23138	153	25	12.29527
4	38	12.0199	54	3	12.18094	104	18	12.23159	154	69	12.29721
5	30	12.0664	55	110	12.1866	105	74	12.23307	155	116	12.29832
6	104	12.08239	56	51	12.18769	106	89	12.23461	156	179	12.30232
7	23	12.08374	57	127	12.18785	107	61	12.23489	157	75	12.30311
8	27	12.08533	58	106	12.19149	108	141	12.23878	158	125	12.30514
9	9	12.08598	59	87	12.19183	109	126	12.24052	159	88	12.30791
10	121	12.08806	60	4	12.19311	110	68	12.24436	160	67	12.3099
11	6	12.08891	61	197	12.19366	111	142	12.24587	161	182	12.31099
12	24	12.08995	62	14	12.19393	112	159	12.24627	162	103	12.311
13	93	12.09057	63	13	12.19535	113	191	12.24641	163	154	12.312
14	48	12.09118	64	107	12.19609	114	136	12.24644	164	194	12.31421
15	91	12.0938	65	56	12.19805	115	178	12.24899	165	131	12.31772
16	20	12.09402	66	145	12.199	116	63	12.25261	166	184	12.31939
17	133	12.10002	67	140	12.1997	117	161	12.25313	167	85	12.32248
18	31	12.1018	68	1	12.19977	118	52	12.25404	168	166	12.32334
19	58	12.10472	69	82	12.19995	119	100	12.25417	169	102	12.32513
20	26	12.1057	70	120	12.20005	120	37	12.25465	170	193	12.3261
21	44	12.10605	71	2	12.20055	121	98	12.2558	171	199	12.32649
22	78	12.1076	72	28	12.20097	122	96	12.2567	172	97	12.32742
23	42	12.11167	73	195	12.20166	123	109	12.25833	173	160	12.32854
24	49	12.11457	74	19	12.20322	124	188	12.25943	174	147	12.32905
25	35	12.11652	75	164	12.20413	125	155	12.26223	175	170	12.32953
26	50	12.12125	76	128	12.20494	126	53	12.26355	176	153	12.33011
27	99	12.12518	77	55	12.20546	127	183	12.26532	177	150	12.33081
28	72	12.1263	78	33	12.20627	128	54	12.26585	178	187	12.33359
29	84	12.12851	79	16	12.20675	129	135	12.26656	179	198	12.33519
30	148	12.12873	80	196	12.20712	130	124	12.26662	180	71	12.33942
31	40	12.1298	81	73	12.2076	131	41	12.268	181	151	12.34268
32	43	12.13092	82	172	12.20793	132	152	12.26908	182	168	12.34396
33	57	12.13265	83	118	12.20816	133	12	12.26971	183	158	12.34432
34	11	12.13266	84	138	12.20854	134	176	12.27027	184	180	12.34515
35	8	12.13321	85	46	12.21009	135	10	12.27219	185	59	12.3456
36	77	12.13644	86	111	12.21032	136	189	12.27226	186	90	12.34621
37	130	12.13917	87	144	12.2104	137	112	12.27233	187	186	12.34728
38	70	12.1419	88	47	12.21173	138	81	12.27273	188	146	12.35113
39	29	12.14399	89	171	12.21275	149	174	12.27273	189	157	12.35747
40	80	12.14632	90	65	12.21397	140	149	12.27416	190	115	12.36179
41	94	12.15128	91	119	12.21452	141	122	12.27493	191	137	12.36437
42	76	12.15187	92	156	12.21736	142	32	12.2752	192	143	12.36616
43	36	12.15568	93	129	12.21791	143	165	12.27582	193	139	12.36629
44	5	12.15643	94	45	12.21819	144	7	12.27665	194	114	12.36831
45	66	12.1583	95	167	12.21867	145	134	12.27908	195	62	12.37101
46	185	12.16356	96	123	12.2208	146	60	12.27965	196	181	12.40196
47	86	12.1653	97	101	12.22286	147	190	12.28106	197	163	12.43148
48	92	12.16575	98	17	12.22462	148	177	12.28113	198	169	12.43463
49	83	12.16598	99	192	12.22492	149	113	12.28593	199	175	12.45786
50	39	12.16905	100	132	12.22856	150	95	12.28921			

Appendix 3. Q5 and Q6 High Voltage Measurements July-August 1998

Q5 HIGH VOLTAGE MEASUREMENTS

VIAL No.	Position	Date	H.V.L.	H.V.R	H.V.G.
16	1	27/07/98	1481.7	1539.7	1337.49
16	1	30/07/98	1481.9	1539.4	1337.51
16	1	14/08/98	1481.8	1539.3	1337.43
16	1	18/08/98	1481.2	1538.3	1337.53
46	2	28/07/98	1481.5	1540.5	1337.5
46	2	30/07/98	1481	1540.5	1337.5
46	2	14/08/98	1482.5	1538.7	1337.42
46	2	17/08/98	1481.5	1538.8	1337.5
73	3	28/07/98	1481.2	1539.8	1337.48
73	3	29/07/98	1480.4	1539.4	1337.5
73	3	14/08/98	1480.9	1538.9	1337.43
73	3	17/08/98	1480.9	1539.3	1337.48
118	4	29/07/98	1481	1539.9	1337.52
118	4	31/07/98	1480.8	1539.3	1337.7
118	4	17/08/98	1481.5	1539.2	1337.5
118	4	18/08/98	1480.4	1539.2	1337.53
144	5	29/07/98	1479.4	1539.9	1337.5
144	5	30/07/98	1479.8	1539.5	1337.49
144	5	18/08/98	1480.2	1538.9	1337.51
144	5	20/08/98	1480.7	1538.4	1337.5
33	41	30/07/98	1480.7	1540.6	1337.56
33	41	03/08/98	1480.1	1540.4	1337.44
33	41	13/08/98	1481.5	1540.1	1337.47
33	41	18/08/98	1480.9	1538.5	1337.53
55	42	28/07/98	1480.1	1539.6	1337.5
55	42	30/07/98	1480.9	1539.1	1337.5
55	42	14/08/98	1480.3	1539.4	1337.44
55	42	17/08/98	1480.5	1543.3	1337.48
111	43	29/07/98	1480.2	1540.3	1337.51
111	43	31/07/98	1480.8	1539.5	1337.51
111	43	17/08/98	1480.5	1539	1337.48
111	43	19/08/98	1480.5	1538.5	1337.53
138	44	29/07/98	1480.6	1539.8	1337.51
138	44	03/08/98	1480.6	1539.9	1337.47
138	44	17/08/98	1480.8	1539.1	1337.5
138	44	25/08/98	1479.7	1538.49	1337.42
172	45	30/07/98	1480.9	1540	1337.5
172	45	03/08/98	1481.9	1539.1	1337.47
172	45	18/08/98	1481.6	1538.4	1337.52
172	45	21/08/98	1482.2	1538.4	1337.55

Q6 HIGH VOLTAGE MEASUREMENTS

VIAL No.	Position	Date	H.V.L.	H.V.R	H.V.G.
1	1	28/07/98	1381.1	1350.2	1193.4
1	1	29/07/98	1380.7	1349.6	1193.46
1	1	13/08/98	1392.2	1360.9	1193.43
1	1	14/08/98	1388.4	1358.2	1193.42
19	2	28/07/98	1382.3	1349.3	1193.46
19	2	29/07/98	1381.6	1348.3	1193.45
19	2	14/08/98	1388.4	1357	1193.43
19	2	19/08/98	1384.5	1352.2	1193.37
82	3	30/07/98	1380.5	1350.2	1193.46
82	3	31/07/98	1378.2	1349.1	1193.43
82	3	21/08/98	1382.5	1350.6	1193.43
82	3	24/08/98	1381.7	1349.8	1193.4
140	4	31/07/98	1379.2	1349.2	1193.47
140	4	03/08/98	1379.6	1347.7	1193.38
140	4	17/08/98	1385.1	1354.6	1193.4
140	4	21/08/98	1382.1	1349.9	1193.4
164	5	03/08/98	1379.6	1350.1	1193.45
164	5	04/08/98	1381	1348.6	1193.5
164	5	17/08/98	1383	1354	1193.42
164	5	18/08/98	1383.5	1352.7	1193.43
2	41	28/07/98	1382.3	1350.2	1193.46
2	41	29/07/98	1381	1349.5	1193.45
2	41	13/08/98	1392.8	1359.2	1193.44
2	41	14/08/98	1388.7	1355.5	1193.42
28	42	29/07/98	1380.8	1350.3	1193.47
28	42	30/07/98	1379.5	1350.3	1193.46
28	42	14/08/98	1388.7	1354.1	1193.43
28	42	20/08/98	1383.7	1354.1	1193.43
120	43	30/07/98	1379.7	1349.7	1193.44
120	43	31/07/98	1379.9	1348.9	1193.4
120	43	17/08/98	1383.2	1353.3	1193.42
120	43	21/08/98	1383	1350.4	1193.4
145	44	31/07/98	1379.5	1348.6	1193.45
145	44	03/08/98	1380.3	1349.5	1193.42
145	44	17/08/98	1382.8	1353.2	1193.42
145	44	18/08/98	1383.1	1351.8	1193.44
195	45	27/07/98	1383.8	1351	1193.45
195	45	03/08/98	1380.4	1350.8	1193.45
195	45	17/08/98	1383.2	1353.8	1193.42
195	45	18/08/98	1383	1353.3	1193.43

High Voltage to the Left Photomultiplier tube (PMT) = H.V.L.

High Voltage to the Right PMT = H.V.R.

Guard High Voltage = H.V.G.

Appendix 4. Comparison of date calculations using Open Window and Balance Point Counting (see McCormac (1992) and Pearson (1983))

Example batch (Q5b) calculated using individual standard data

Background (BGD) counts per minute (CPM) Values

	Q5_BGD0399_V33 CPM	Q5_BGD0399_V33 CPM	
Restricted Window	Channel 218-484	0.357	0.357
Open Window	Channel 108-568	0.76	0.76

Oxalic 1950 Value Calculation

Q5_OX0399_V16

	Start Weight	Mean Counting Weight	CPM	Mass Spec. Value ($\delta^{13}\text{C}$ ‰)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Synthesis factor (Syn. fac.)	CPM/g	CPM/g corr. for Mass. Spec.	CPM/g corr. for 1950 Value
Restricted Window	Channel 218-484	2.62783	32.507	-17.723	0.985446	0.357	32.12678	12.26946	12.09088896	9.018594078
Open Window	Channel 108-568	2.62783	37.791	-17.723	0.985446	0.76	37.00778	14.13355	13.92784956	10.38878299

Radiocarbon Age Calculation

	Channel	Mean Counting Weight	% sample	CPM	Mass Spec. Value ($\delta^{13}\text{C}$ ‰)	Mass Spec. Correction Factor	BGD CPM	CPM corr. for Bgd and Syn.fac.	CPM/g	CPM/g corr. for Mass. Spec.	Oxalic 1950 Value	Radiocarbon Age BP
Restricted Window	Channel 218-484											
	UB-4405	2.62439	100	20.661	-25.733	1.001466	0.357	20.28078	7.727807	7.739136189	9.018594078	1229
	UB-4406	2.62622	100	20.678	-25.903	1.001806	0.357	20.29778	7.728896	7.742853908	9.018594078	1225
Open Window	Channel 108-568											
	UB-4405	2.62439	100	23.974	-25.733	1.001466	0.76	23.35978	8.901032	8.914081153	10.38878299	1230
	UB-4406	2.62622	100	24.143	-25.903	1.001806	0.76	23.37278	8.89978	8.915852914	10.38878299	1228