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GLASGOW UNIVERSITY RADIOCARBON MEASUREMENTS IX

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INTRODUCTION

The following date list presents results obtained during 1975-1977. The facilities at Glasgow have been further developed and include a gas counting system utilizing CO_2 as counting material and two liquid scintillation counting laboratories based on synthesis of benzene. The results presented here were obtained using the liquid scintillation system only. Sample pretreatment varied according to individual sample type, whether wood or charcoal. The majority of samples were of preserved wood. After manual removal of gross contamination, samples of wood were finely chopped and digested in boiling, 2M KOH solution. The dehumified wood was then separated by filtration, thoroughly washed with distilled water and dilute hydrochloric acid, and then bleached in a solution of $NaClO_2/HCl$ at 80°C for 48 hours. Pure, white wood cellulose was obtained by filtration and washed with a large volume of distilled water. The cellulose, typically 40% by weight of the original material, was then dried overnight in an oven at 80°C. Samples of charcoal were closely examined for non-contemporaneous contamination and then subjected to successive treatments with hot, dilute (1M) KOH and HCl, respectively. The remaining charcoal was then dried overnight at 80°C.

Pretreated samples were converted to benzene for counting using standard procedures. In summary, samples were converted to CO_2 by combustion in a stainless steel pressure reaction vessel. A sample of CO_2 was removed for subsequent mass spectrometric assay. CO_2 was converted to C_2H_2 by reaction with molten lithium in a second stainless steel reaction vessel. When cool, the product lithium carbide was hydrolyzed with freshly distilled water, yielding acetylene. Finally benzene was produced by cyclotrimerization over an activated catalyst (KC Perl Katalysator Neu).

Samples were counted using a Packard Tricarb Series 3330 Liquid Scintillation Spectrophotometer optimized for ^{14}C detection. A standard 10ml counting geometry was adopted, the scintillation cocktail comprising 8ml synthesized benzene and 2ml of a PPO/POPOP toluene-based scintillation solution. (Where insufficient benzene was synthesized, samples were diluted to the standard volume with "dead" spectroscopic grade benzene.) The long-term stability of the counting system was assessed by continuous monitoring of background and NBS oxalic acid modern standard samples. An excellent degree of stability was indicated. Count rates for NBS oxalic acid and background samples were measured as 63.40 ± 0.04 cpm and 7.44 ± 0.01 cpm, respectively. Observed sample and standard activities were corrected for the effects of quenching by means of a sample channels ratio method. Measurement of the stable carbon isotope

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enrichment, $\delta^{13}C$, was performed using a VG Micromass 602B stable isotope mass spectrometer to a precision of $\pm 0.05‰$ ($\pm 1\sigma$). The uncertainty quoted on radiocarbon age determinations reported here represents the $\pm 1\sigma$ counting errors alone and does not include contributions from extraneous experimental variability.

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SAMPLE DESCRIPTIONS

I. INTERCALIBRATION SAMPLES

Intercalibration samples were analyzed at regular intervals during 1975-1977. In addition to samples subm specifically for intercalibration purposes, a number of "check" analyses was performed on selected tree-ring samples. Results of check analyses will be reported with appropriate tables in subsequent secs of the text. (The designation "GUA" refers to analyses on the Glasgow CO_2 gas counting system which have yet to be assigned GU- code numbers.)

GU-656. **166 ± 42**

Hitchcock wood. *Comment*: LJ-3451, 142 ± 68; LJ-3452, 221 ± 89.

GU-657. **$\Delta^{14}C = 5.01 \pm 5.62‰$**

Charcoal. *Comment*: LJ-3448, $\Delta^{14}C = -3.8 \pm 6.1‰$.

GU-658. **3505 ± 58**

Pine wood. *Comment*: LJ-3453, 3793 ± 61; GUA, 3551 ± 58.

GU-659. Penzance, Cornwall **4093 ± 50**

Wood. *Comment*: sample previously described and reported under SRR-714, 4278 ± 50. Also GUA, 4025 ± 67.

GU-660. West Runton 6 **26,450 ± 420**

Wood. *Comment*: sample previously described and reported under SRR-225, -228, >47,500; GrN-6819, >54,200; GrN-6892, >47,400. Also GUA, 21,732 ± 532.

GU-661. West Runton 6 **26,400 ± 480**

As GU-660.

General Comment: discrepancy between age determinations on West Runton 6 sample for Glasgow systems, both liquid scintillation and gas counting, and those at SRR and GrN, suggests sample contamination (by ~4% modern carbon) at some point between time of assay at SSR and GrN and more recent measurements at Glasgow.

Considered along with "check" samples reported later in the text, replicate measurement and accumulated NBS oxalic acid standard samples, samples GU-656-661 imply satisfactorily accurate measurement.

II. REPLICATE ANALYSIS STANDARDIZATION

Two replicate analysis "standards" were adopted for this study. A full description of relevant procedures may be found in Campbell (1977).

Replicate Series 1

Bulk homogenized wood (*Pinus*) from Ynyslas submerged forest exposure (52° 29′ N, 4° 4′ W). Sample also subjected to sequential growth-increment analysis (see GU-711, -718, below). Coll by A Heyworth.

Sample no.	Radiocarbon age	$\delta^{13}C$‰
GU-669	4825 ± 52	−24.0
GU-670	5012 ± 41	−24.4
GU-671	4094 ± 39	−24.2
GU-672	5004 ± 39	−23.7
GU-673	4819 ± 45	−24.1
GU-674	5003 ± 37	−24.2
GU-675	4823 ± 42	−23.8
GU-676	4791 ± 40	−24.1
GU-677	4752 ± 33	−24.0
GU-678	4930 ± 37	−23.9
GU-679	4873 ± 33	−23.7
GU-680	4765 ± 35	−23.9
GU-681	4863 ± 36	−23.7
GU-682	4899 ± 34	−23.7
GU-683	4770 ± 37	−23.9
GU-684	4866 ± 35	−23.8
GU-685	4747 ± 42	−23.9
GU-686	4887 ± 30	−24.0
GU-687	4771 ± 35	−24.0
GU-688	4968 ± 29	−24.1
GU-689	4788 ± 28	−24.4
GU-690	4933 ± 29	−26.1
GU-691	4839 ± 28	−23.9
GU-692	4739 ± 33	−24.2

Comment: sample of replicate standard analyzed by SRR, 4926 ± 52 (Harkness, written commun).

Replicate Series 2

Rice grain.

Sample no.	$\delta^{14}C$‰	$\delta^{13}C$‰	$\Delta^{14}C$‰
GU-693	416.44 ± 3.41	−23.9	413.32 ± 3.42
GU-694	397.40 ± 3.36	−24.0	394.61 ± 3.36
GU-695	415.39 ± 3.36	−24.3	413.41 ± 3.36
GU-696	418.22 ± 3.51	−25.4	419.36 ± 3.50
GU-697	401.96 ± 3.42	−25.0	401.96 ± 3.42
GU-698	422.33 ± 3.60	−23.8	418.92 ± 3.61
GU-699	404.38 ± 3.42	−22.8	398.20 ± 3.44
GU-700	391.52 ± 3.63	−24.1	389.02 ± 3.64
GU-701	397.24 ± 3.42	−24.0	394.45 ± 3.43
GU-702	400.97 ± 3.39	−23.8	397.43 ± 3.40

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III. ARCHAEOLOGIC SAMPLES

South Cadbury series

Samples from site of Cadbury Castle, England (51° 01′ N, 2° 32′ W). Samples coll by L Alcock, Dept Archaeol, Univ Glasgow.

GU-645. SC/K 659-i **1814 ± 31**
$\delta^{13}C = -24.6‰$

Wood charcoal. Twig remains from guard chamber, context SC/K 659. *Comment*: sample material also analyzed as SRR-693, 1845 ± 45 (AD 105).

GU-646. SC/K 659-ii **1961 ± 27**
$\delta^{13}C = -25.8‰$

Wood charcoal assoc with large timbers from roof of guard chamber, context SC/K 659.

GU-647. SC/K 659-iii **1839 ± 26**
$\delta^{13}C = -25.2‰$

Wood charcoal assoc with large timbers from roof of guard chamber, context SC/K 659.

GU-648. SC/K 659-iv **2214 ± 43**
$\delta^{13}C = -24.8‰$

Wood charcoal assoc with large timbers from roof of guard chamber, context SC/K 659.

GU-649. SC/K 659-v **1949 ± 26**
$\delta^{13}C = -24.1‰$

Carbonized grain from floor of guard chamber, context SC/K 659. *Comment*: sample material also analyzed as SRR-691, 1776 ± 50 (AD 174).

GU-650. SC/K 659-vi **1765 ± 47**
$\delta^{13}C = -24.3‰$

Carbonized grain from floor of guard chamber, context SC/K 659. *Comment*: sample material also analyzed as SRR-692, 1666 ± 50 (AD 284).

GU-651. SC/K 747 **1825 ± 48**
$\delta^{13}C = -23.7‰$

Wood charcoal assoc with structural timbers of gateway, context SC/K 747.

General Comment: South Cadbury series marks Roman sack of Cadbury Castle, an event placed in mid-first century AD, based on historico-archaeol evidence. Previous date on material from closely related level yielded age, AD 444 ± 40 (SRR-444), clearly at variance with archaeol evidence. This further study (Alcock *et al*, ms in preparation) shows improved concordance, although radiometric measurements still indicate more recent ages than theoretical prediction.

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Los Tayos series

Samples coll during 1976 British-Ecuadorian Expedition to Los Tayos Caves.

GU-652. OM-S6 **1012 ± 66**
$\delta^{13}C = -28.6‰$

Wood charcoal assoc with "chimney-burial" pottery, Teniente Ortiz, Morona Santiago, Ecuador (3° 3′ S, 78° 2′ W).

GU-653. OM-ST1-4 **235 ± 36**
$\delta^{13}C = -27.6‰$

Wood charcoal, Teniente Ortiz.

GU-654. OM-ST1-5 **125 ± 60**
$\delta^{13}C = -27.6‰$

Wood charcoal, Teniente Ortiz.

GU-793. MS-LT-1 **$\delta^{14}C = 2.58 \pm 1.38‰$**

Soil sample at 2.3m depth in guano pile, main Los Tayos cave.

GU-792. Castle Point, Banffshire **3107 ± 303**
$\delta^{13}C = -25.1‰$

Wood sample from early occupation site on Castle Point (57° 41′ 7″ N, 2° 17′ 26″ W).

IV. TREE RING SAMPLES

Past variations in natural radiocarbon content of the atmosphere were studied through ^{14}C assay of "floating" chronologies constructed from tree-ring records of preserved wood located in submerged forest sites along the west coast of England and Wales (Campbell, 1977). These forests provide an ideal source of material for dendrochronologic and radiocarbon studies. Individual exposures are mainly of oak, with some pine and, to a lesser extent, alder, birch, and yew. Growth patterns are extremely sensitive and individual rings are wide giving plentiful dating material and higher sampling frequency than in previous studies (Suess, 1970; 1976; Pearson *et al*, 1977). Dendrochronologic measurements are being made by A Heyworth, Univ College, Wales at Aberystwyth.

Preliminary dates on samples coll at several locations have indicated wide range of ages available in submerged forest exposures.

GU-662. Clarach, Dyfed **5592 ± 143**

Wood sec, Clarach-1 (*Quercus*) from submerged forest exposure (52° 26′ N, 4° 4′ W). Coll by A Heyworth.

GU-663. Stolford, Somerset **4443 ± 199**

Wood sec Stolford-1 (*Quercus*) from submerged forest exposure (51° 12′ N, 3° 6′ W). Coll by A Heyworth.

GU-664. Morecambe, Lancashire **7544 ± 306**

Wood sec Morecambe-1 (*Betula*) from submerged forest exposure (54° 04′ N, 2° 54′ W). Coll by A Heyworth.

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GU-665. Llanaber, Gurynedd **734 ± 52**

Wood sec Llanaber-1 (*Larix*) from submerged forest exposure (52° 44′ N, 4° 4′ W). Coll by A Heyworth.

GU-666. Alt-Mouth, Lancashire **4351 ± 46**

Wood sec Alt-Mouth-1 (*Quercus*) from submerged forest exposure (53° 32′ N, 3° 4′ W). Coll by A Heyworth.

GU-667. Newton, Powys **885 ± 83**

Wood sec Newton-1 (*Quercus*) from gravel bed on River Severn (52° 31′ N, 3° 19′ W).

GU-668. Rheidol, Dyfed **647 ± 136**

Wood sec Rheidol-1 (*Quercus*) from gravel bed on River Rheidol (52° 23′ N, 3° 58′ W).

Borth-1 tree-ring sequence, Borth, Dyfed series

Wood sec Borth-1 (*Pinus*) comprising 90 growth rings. From submerged forest exposure (52° 29′ N, 4° 4′ W). Coll by A Heyworth.

Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C$‰
GU-703	0-20	5167 ± 42	−24.1
GU-704	21-30	5249 ± 36	−24.1
GU-705	31-40	5277 ± 37	−24.3
GU-706	41-50	5540 ± 37	−23.9
GU-707	51-60	5240 ± 36	−23.9
GU-708	61-70	5314 ± 40	−23.9
GU-709	71-80	5124 ± 36	−23.6
GU-710	81-90	5174 ± 84	−23.9

Borth-4 tree-ring sequence, Borth, Dyfed series

Wood sec Borth-4 (*Quercus*) comprising 155 growth rings. From submerged forest exposure (52° 29′ N, 4° 4′ W). Coll by A Heyworth.

Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C$‰
GU-742	−15-+10	3917 ± 84	−26.1
GU-743	11-30	3781 ± 95	−25.7
GU-744	31-40	3783 ± 58	−27.0
GU-745	41-50	4027 ± 35	−27.1
GU-746	51-60	3798 ± 30	−25.5
GU-747	61-70	3832 ± 31	−24.1
GU-748	71-80	3705 ± 37	−24.8
GU-749	81-90	3751 ± 61	−26.1
GU-750	91-100	3835 ± 29	−24.5
GU-751	101-110	3838 ± 46	−24.9
GU-752	111-120	3856 ± 33	−24.7
GU-753	121-130	3812 ± 31	−24.6
GU-754	131-140	3828 ± 32	−24.5

Comment: GU-746 was also analyzed by SRR, 3796 ± 51 (Harkness, written commun).

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Borth-6 tree-ring sequence, Borth, Dyfed series

Wood sec Borth-6 (*Quercus*) comprising 270 growth rings. From submerged forest exposure (52° 29′ N, 4° 4′ W). Coll by A Heyworth.

Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C‰$
GU-719	1-10	4419 ± 49	−25.7
GU-720	11-22	4284 ± 47	−25.3
GU-721	23-40	4029 ± 104	−25.0
GU-722	41-69	4052 ± 35	−24.2
GU-723	70-80	4219 ± 46	−24.1
GU-724	81-90	3958 ± 36	−25.2
GU-725	91-100	3825 ± 69	−26.1
GU-726	101-112	3762 ± 54	−26.1
GU-727	113-120	3968 ± 40	−23.1
GU-728	121-130	3872 ± 42	−24.9
GU-729	131-140	4190 ± 37	−25.4
GU-730	141-150	3960 ± 46	−24.3
GU-731	151-160	3738 ± 43	−25.7
GU-732	161-170	4120 ± 45	−25.3
GU-733	171-180	4189 ± 87	−24.4
GU-734	181-190	3961 ± 47	−24.0
GU-735	191-200	4006 ± 33	−24.0
GU-736	201-210	3984 ± 60	−24.2
GU-737	211-220	3999 ± 26	−24.3
GU-738	221-230	4198 ± 44	−24.3
GU-739	231-240	4034 ± 45	−25.7
GU-740	241-250	4068 ± 45	−25.0
GU-741	251-270	3957 ± 64	−24.0

Comment: GU-730, -731 were also analyzed by SSR, yielding respective ages, 4016 ± 53; 3958 ± 52 (Harkness, written commun).

Ynyslas-1 tree-ring sequence, Ynyslas, Dyfed series

Wood sec Ynyslas-1 (*Pinus*) comprising 80 growth rings. From submerged forest exposure (52° 29′ 30″ N, 4° 4′ W). Coll by A Heyworth.

Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C‰$
GU-711	−10-+10	5106 ± 48	−24.5
GU-712	0-10	4935 ± 89	−24.7
GU-713	11-20	5009 ± 52	−23.9
GU-714	21-30	4974 ± 39	−24.1
GU-715	31-40	4946 ± 46	−24.3
GU-716	41-50	5035 ± 46	−24.2
GU-717	51-60	4924 ± 40	−23.6
GU-718	61-70	4930 ± 48	−23.3

Stolford-4 tree-ring sequence, Stolford, Somerset series

Wood sec Stolford-4 (*Quercus*) comprising 80 growth rings. From submerged forest exposure (51° 12′ N, 3° 6′ W). Coll by A Heyworth.

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Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C$‰
GU-755	1-10	4733 ± 34	−24.1
GU-756	11-20	4803 ± 36	−24.0
GU-757	21-30	4718 ± 31	−24.4
GU-758	31-40	4645 ± 41	−24.2
GU-759	41-50	4490 ± 47	−23.9
GU-760	51-60	4715 ± 31	−24.1
GU-761	61-70	4778 ± 42	−23.1
GU-762	71-80	4707 ± 32	−22.8

Stolford-5 tree-ring sequence, Stolford, Somerset series

Wood sec Stolford-5 (*Quercus*) comprising 302 growth rings. From submerged forest exposure (51° 12′ N, 3° 6′ W). Coll by A Heyworth.

Sample no.	Growth rings	Radiocarbon age	$\delta^{13}C$‰
GU-763	−12-+10	5398 ± 79	−23.9
GU-764	11-20	5298 ± 66	−23.8
GU-765	21-30	5201 ± 55	−23.6
GU-766	31-40	5285 ± 45	−23.6
GU-767	41-50	5399 ± 40	−23.3
GU-768	51-60	5269 ± 40	−23.1
GU-769	61-70	5258 ± 60	−23.2
GU-770	71-80	5186 ± 45	−22.9
GU-771	81-90	5382 ± 43	−23.0
GU-772	91-100	5377 ± 41	−23.4
GU-773	101-110	5220 ± 39	−23.6
GU-774	111-120	5169 ± 53	−23.9
GU-775	121-130	5204 ± 72	−23.7
GU-776	131-140	5094 ± 51	−23.4
GU-777	141-150	5067 ± 81	−24.0
GU-778	151-160	5092 ± 70	−23.8
GU-779	161-170	4840 ± 127	−23.5
GU-780	171-180	5020 ± 104	−24.1
GU-781	181-190	5039 ± 61	−23.7
GU-782	191-200	5092 ± 67	−23.6
GU-784	211-220	5007 ± 61	−23.6
GU-785	221-230	5015 ± 68	−23.4
GU-786	231-240	4844 ± 97	−23.8
GU-787	241-250	4958 ± 54	−23.8
GU-788	251-260	4783 ± 67	−24.3
GU-789	261-270	4775 ± 124	−29.9
GU-790	271-280	4908 ± 68	−23.4
GU-791	281-290	4831 ± 67	−24.7

General Comment: results of sequential growth-increment analyses clearly indicate that short-term fluctuations in natural radiocarbon content of the atmosphere have occurred over the past millennia. Variations of 2 to 3% over several decades are comparable with those reported by Campbell *et al* (1978) from similar measurements on a "floating" tree-ring chronology dating from early 3rd millennium BP (radiocarbon years). It is firmly believed that detection of these variations has been facilitated by high sampling frequency employed in this study.

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References

Campbell, J A, 1977, Past variations in natural radiocarbon as recorded in UK wood: PhD thesis, Univ Glasgow.

Campbell, J A, Baxter, M S, and Harkness, D D, 1978, Radiocarbon measurements on a floating tree-ring chronology from north-east Scotland: Archaeometry, v 20, p 33-38.

Pearson, G W, Pilcher, J R, Baillie, M G L, and Hillam, J, 1978, Absolute radiocarbon dating using a low altitude European tree-ring calibration: Nature, v 270, p 25-28.

Suess, H E, 1970, Bristlecone pine calibration of the radiocarbon time-scale 5200 BC to the present, *in*: Olsson, I U (ed), Radiocarbon variations and absolute chronology, Nobel symposium 12th, Proc, Uppsala, 1969, p 303-312, New York, John Wiley.

——— 1976, 9th internatl radiocarbon dating conf, Proc, Los Angeles, in press.