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FLORIDA STATE UNIVERSITY RADIOCARBON DATES I

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Laboratory construction was begun in December 1964 and routine dating was in progress by late February 1965. The primary purpose of this facility is to assist in marine geological studies and the archaeological chronologies of the region. Dating is carried out by utilizing the techniques of liquid scintillation described by Noakes *et al.* (1965), wherein the carbon of the sample to be dated is converted to benzene and the natural radioactivity detected in a liquid scintillation spectrometer.

After sample pretreatment to remove non-contemporaneous carbon, the sample is chemically converted as follows:

$$\text{Sample} \rightarrow CO_2 \rightarrow C_2H_2 \rightarrow C_6H_6$$

Conversion to CO_2 is a fairly standard procedure in all laboratories and therefore will not be discussed. Conversion of $CO_2 \rightarrow C_2H_2$ via Li_2C_2 is an adaption of the technique used by Barker (1953), which gives consistent chemical yields of 95-98%. Conversion of acetylene to benzene is carried out through the action of a vanadium-alumina catalyst which gives yields of 85-92% of very pure benzene.

Noakes, Kim and Stipp (1965) undertook mass spectrometric studies at the Oak Ridge National Laboratories on the possible isotopic fractionation in the conversion of acetylene to benzene prepared both by the original diborane-activated silica-alumina catalyst described by Noakes *et al.* (1963) and with the new high-valence metal oxide catalysts described by Noakes, Kim and Akers (1965). In this study he found that even over a large range of induced experimental yields (30-98%) there was no isotopic fractionation. This had previously been indicated through reproducibility studies (Stipp *et al.*, 1962; Noakes *et al.*, 1964; Tamers *et al.*, 1964; McDowell and Ryan, 1965), and through statistical comparison techniques (Tamers and Pearson, 1965).

Barker's (pers. comm., 1962) acetylene purification method, using columns filled with glass beads coated with concentrated phosphoric acid and 50% KOH, has insured against quenching by removing nitrogen-hydrogen and sulphur products.

Radon, if initially present, should be removed during the high temperature evacuation carried out on the hot lithium carbide. Apparently this step is quite effective as no radon has ever been seen to contaminate any of the counting solutions.

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Detection is carried out in an ANS, Inc. automatic two-channel Liquid Scintillation Spectrometer. It has been shown previously by McDowell (pers. commun., 1964) and Pietig and Scharpenseel (1964) that supplemental shielding with mercury in liquid counting has a significant effect in reducing background levels. Therefore, in cooperation with ANS, Inc., an experiment at Florida State University using a two-in. shield of triple-distilled mercury in place of the standard lead shield was carried out with the result that backgrounds were lowered by approx. 20%. The standard vial size chosen is 5cc which gives a background of 7.41 counts/min and a modern standard rate (age-corrected 1860 wood) of 34.87 counts/min yielding an age range of 44,000 yr for a two-sigma deviation and 48 hour counting period. For older samples a 15 cc vial is used with a background rate of 12.82 counts/min and a modern rate of 111.16 counts/min, with an extension of age range to 51,800 yr on the same criterion.

The possibility of sample quenching is closely monitored both by the channels ratio and the automatic external standardization method employing a Cs^{137} gamma source ($5_{\mu c}$). Both methods are sensitive in detecting any sample or electronic discrepancies to less than one percent.

The modern standard, once accurately determined, is checked daily thereafter with a "hot" reference standard (Pearson, pers. commun., 1964; Noakes *et al.* 1965). This technique has allowed nearly continuous monitoring of the modern rate while at the same time eliminating the necessity for frequent and time-consuming preparation of modern samples.

Data calculation is done by a Fortran II program which also analyzes continuous instrument performance from the frequent-interval data print-out. Arrangements are now being made to print the raw data onto a coded punch tape to further facilitate handling.

Dates contained in the following partial list were calculated using the Libby half-life of 5570 yr and reported with their counting error uncertainty of 1 standard deviation. Because of the lack of suitable mass-spectrometric facilities no corrections are made for natural C^{13}/C^{12} ratio fluctuations. All collectors and submitters of samples from Florida State University, Tallahassee, Florida, unless stated otherwise.

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SAMPLE DESCRIPTIONS

I. CHECK SAMPLES

Florida number	Florida date	Other number	Other date	Reference
FSU-1a FSU-1b FSU-1c	2320 ± 100 2360 ± 80 2380 ± 80	ML-86	2025 ± 55	Miami I-III
FSU-2	9880 ± 230	ML-153B	9900 ± 105	Miami I-III
FSU-3	11,245 ± 450	C -800 TX-44 L -6980	10,856 ± 410 10,700 ± 210 11,840 ± 100	Chicago IV Texas II Broecker and Farrand (1963)

II. GEOLOGIC SAMPLES

A. South-East U. S.

Tampa Bay series, Florida

Oyster shell and meat from various localities within Tampa Bay. Oysters were living at time of collection and were measured to provide indication of any significant difference in natural fractionation between shell and flesh fractions of modern oysters. Results were correlated with pH, salinity and temperature data. Coll. May 1965 by C. H. Solomon, U. S. Dept. of Interior; subm. by H. S. Rydell.

Number	Name	Material	N Lat	W Long	$C^{14}\%$ > Mdn
FSU-12.	Big Bend	Meat	27° 46′ 18″	82° 26′ 0″	21.3 ± 0.23
FSU-16.	Big Bend	Shell	27° 46′ 18″	82° 26′ 0″	22.7 ± 0.30
FSU-13.	Piney Pt.	Meat	27° 38′ 36″	82° 33′ 40″	20.3 ± 0.37
FSU-17.	Piney Pt.	Shell	27° 38′ 36″	82° 33′ 40″	23.6 ± 0.32
FSU-14.	Rocky Pt.	Meat	27° 57′ 42″	82° 54′ 36″	22.5 ± 0.54
FSU-18.	Rocky Pt.	Shell	27° 57′ 42″	82° 54′ 36″	25.5 ± 0.35
FSU-15.	Skyway	Shell	27° 40′ 18″	82° 41′ 0″	24.4 ± 0.30

Appalachicola Bay series, Florida

Samples are of recent and Pleistocene surface and subsurface wood and shell material collected between Cape San Blas and Alligator Harbor, Florida. Dates represent part of study of coastal evolution of Appalachicola delta region of NW Florida with respect to late Pleistocene-Holocene sealevel fluctuations. Coll. 1964 and early 1965. Subm. 1965 by J. Schnable.

560 ± 100

FSU-23. St. George–1 **A.D. 1390**

Wood from taproot stump of pine tree in growth position and now awash (intertidal) on present beach. Coll. ½ mi SW of New Pass, St. George Island, Florida (29° 36′ 30″ N Lat, 84° 58′ 20″ W Long).

4610 ± 625

FSU-24. Royal Bluff **2660 B.C.**

Sandy fresh-water peat from intertidal zone coll. W of Carrabelle on mainland shore approx. 1 mi E of Royal Bluff, Florida (29° 48′ 6″ N Lat, 84° 44′ 18″ W Long).

1390 ± 175

FSU-25. Alligator Harbor **A.D. 560**

Wood chips from peat layer ca. 1 ft above MSL from E shore of Alligator Harbor, Florida (29° 54′ 0″ N Lat, 84° 22′ 0″ W Long).

350 ± 120

FSU-26. Alligator Harbor **A.D. 1600**

Wood from taproot stump of pine tree in growth position and now awash (intertidal) on present beach. Coll. from SW Cape on Alligator Spit (29° 53′ 30″ N Lat, 84° 22′ 30″ W Long).

$40{,}340^{+1630}_{-1350}$

FSU-28. St. George—3 **38,390 B.C.**

Log, probably driftwood, at 41 ft below MSL in continuously cored section of Pleistocene sediments. Boring site in old dune area on St. George Island, Florida (29° 39′ 42″ N Lat, 84° 51′ 12″ W Long).

FSU-29. St. George—4 **>30,000**

Wood fragments, probably driftwood, at 36 ft below MSL in continuous cored section of Pleistocene sediments. Boring site on present beach, St. George Island, Florida (29° 39′ 36″ N Lat, 84° 51′ 12″ W Long).

FSU-30. St. George—5 **>27,620**

Wood fragments, probably driftwood, at 38 ft below MSL from a boring site in old dune area on St. George Island, Florida (29° 39′ 42″ N Lat, 84° 52′ 24″ W Long).

$30{,}700^{+2760}_{-3050}$

FSU-31. St. George—6 **28,750 B.C.**

Wood fragments, probably driftwood, at 37.5 ft below MSL in a continuously cored section of Pleistocene sediments. Boring site on present beach, St. George Island, Florida (29° 39′ 36″ N Lat, 84° 51′ 12″ W Long).

3780 ± 330

FSU-32. Lanark Village **1830 B.C.**

Wood fragments in a subsurface sandy peat from ca. 5 ft below MSL. Boring site on bayshore, Lanark, Florida (29° 52′ 51″ N Lat, 84° 35′ 48″ W Long).

4100 ± 110

FSU-33. St. George—7 **2150 B.C.**

Oyster shells (*Crassostrea virginica*), from bay deposit 18 ft below MSL. Boring site on bayside of St. George Island ca. 1 mi W of ferry landing (20° 39′ 48″ N Lat, 84° 52′ 0″ W Long).

4370 ± 420

FSU-34. St. George—8 **2420 B.C.**

Reworked oyster shells (*Crassostrea virginica*), from barrier island sand deposit 12.5 ft below MSL. Boring site on bay side of St. George Island ca. 1 mi W of ferry landing (same location as FSU-33). *Comment:* although FSU-33 and FSU-34 appear to be reversed, they are statistically indistinguishable.

Comment (J.S.): dates in the 30,000 to 40,000 yr B.P. range indicate possibility of a relatively high stand of sea during mid-Wisconsin. Dates are on wood believed to be driftwood occurring in subsurface Pleistocene sediments. Dates less than 5000 yr B.P. on wood fragments and oyster shells give some indication of Holocene sealevel rise. More dates concerning this study will be published in the future.

Alligator-Horeshoe Creek series

Fossil wood buried at various depths in recent soils from the drainage area of Alligator and Horseshoe Creeks, in Gulf Co., Florida. These samples, plus others to be dated, should indicate rate of soil formation in the respective areas. Coll. 1965 by B. Williamson of Gulfland Timber Co., Florida; subm. 1965 by W. F. Tanner.

1520 ± 80

FSU-59. Gulf Co.—1 **A.D. 430**

From hillside in Gulf Co., Florida (30° 6′ 54″ N Lat, 85° 15′ 16″ W Long), at 29 in. depth.

520 ± 140

FSU-60. Gulf Co.—2 **A.D. 1430**

From hillside in Gulf Co., Florida (30° 3′ 34″ N Lat, 85° 15′ 17″ W Long), at 10 in. depth.

480 ± 145

FSU-61. Gulf Co.—3 **A.D. 1470**

From hilltop in Gulf Co., Florida (30° 3′ 22″ N Lat, 85° 15′ 9″ W Long), at 11 in. depth.

2630 ± 135

FSU-62. Gulf Co.—4 **680 B.C.**

From flood plain in Gulf Co., Florida (30° 2′ 5″ N Lat, 85° 15′ 2″ W Long), at 25 in. depth.

Comment (W.F.T.): FSU-61, only hilltop sample of the four, yielded fastest rate of soil formation (1 in./43 yr). FSU-62, the only floodplain sample, yielded slowest rate (1 in./106 yr). The other two (hillside samples) taken ca. 4 mi apart, produced essentially identical rates of soil formation (1 in./52 yr). This was unexpected, as hilltop soils normally develop less rapidly than floodplain material. Several explanations are being considered pending further dates.

B. Antarctic

Eltanin series

Core samples of foraminiferal ooze (*Globigerina parhyderma*) taken during U.S.N.S. Eltanin Cruise 10. These were used to compare Th^{230}/Th^{232} age method with C^{14} method. Coll. Nov. 1964 by I. Zemmels; subm. Mar. 1965 by C. Holmes.

18,240 ± 1050

FSU-46. Pacific Basin **16,290 B.C.**

From E edge of SE Pacific Basin (64° 5′ 30″ S Lat, 75° 19′ 42″ W Long). Sample taken from Core No. 10-15, 0 to 10 cm from top of core.

22,460 ± 925

FSU-47. Pacific Rise **20,510 B.C.**

From E edge of SE Pacific Rise (64° 10′ 30″ S Lat, 75° 18′ 0″ W Long). Sample taken from Core No. 10-14, 5 to 15 cm from top of core.

23,940 ± 925

FSU-48. Pacific Basin **21,940 B.C.**

From E edge of SE Pacific Basin (64° 5′ 30″ S Lat, 75° 19′ 32″ W Long). Sample taken from Core No. 10-15, 20 to 30 cm from top of core.

FSU-49. Drake Passage **>23,600**

From middle of Drake Passage (60° 2′ 0″ S Lat, 64° 54′ 0″ W Long). Sample taken from Core No. 4-14, 85 to 95 cm from top of core. *Comment:* dates from the two methods (Holmes, 1962) were found to differ by a factor of 2, C^{14} ages being younger.

11,960 ± 265

FSU-57. Pacific Ridge **10,010 B.C.**

Sample of *Globigerina pachyderma, Globigerina bulloides,* and *Globigerina inflata,* off Pacific Antarctic Ridge (54° 25′ 0″ S Lat, 129° 37′ 36″ W Long), at 1890 fm depth. Taken from trigger core No. 24, 0 to 19 cm from top of core on U.S.N.S. Eltanin, Cruise 13. Coll. 1964 by M. Boeuf; subm. 1965 by H. Goodell.

9730 ± 255

FSU-50. Scotia Ridge **7780 B.C.**

Unidentified coral dredged from a seamount (59° 56′ 8″ S Lat, 34° 41′ 1″ to 34° 32′ 7″ W Long), at 620 fm depth. Coll. 1964 by S. Koster; subm. 1965 by H. Goodell. *Comment:* gives date B.P. that coral lived and

since no living coral has been found on Sars Bank in this locality at present time it gives a max time for sealevel rise.

III. ARCHAEOLOGIC SAMPLES

1150 ± 150

FSU-63. Basin Bayon West, Florida **A.D. 800**

Charcoal from ramp fill of primary mound at Site WL 13, Walton Co., Florida (30° 29′ 59″ N Lat, 86° 14′ 30″ W Long). Site excavated by Moore (1901) and assigned to Weeden Island I Phase by Willey (1949). Coll. 1958 and subm. 1964 by W. Lazarus, Fort Walton Beach, Florida. *Comment:* date closely approximates midrange of Weeden Island phase.

945 ± 140

FSU-65. Butcherpen Mound, Florida **A.D. 1005**

Charcoal lumps in Pit 1, Level 2, of Site Sa29 in association with E side sherd deposit of mound (30° 23′ 30″ N Lat, 87° 7′ 15″ W Long). *Comment:* may date Weeden Island II Phase on this part of Gulf coast. Coll. 1961 and subm. 1964 by W. Lazarus.

3085 ± 130

FSU-64. Alligator Lake, Florida **1135 B.C.**

Charcoal from pit in Area A of Site W1 29, Walton Co., Florida. Site is reported by Lazarus (1965) (30° 20′ 10″ N Lat, 86° 12′ 10″ W Long). Coll. and subm. by W. Lazarus. *Comment:* sample was in association with a vessel of Alexanders type.

210 ± 85

FSU-68. Buck Site, Alabama **A.D. 1740**

Charred wood from a feature containing oyster shells and cultural material of Fort Walton Phase at Site 1 Ba 56, Baldwin Co., Alabama (30° 17′ N Lat, 87° 43′ W Long). Coll. 1965 by D. White; subm. 1965 by D. Phelps. *Comment:* date seems late for Fort Walton Phase, but duration of phase is little known at present in this area.

Tucker Site series, Florida

Charred material from surfaces and interiors of vessel fragments obtained from eroding beach component of Tucker Site (Fr 4), Florida (29° 55′ 15″ N Lat, 84° 22′ 31″ W Long). Burial mounds on site were excavated by Moore (1902). Willey (1949) records the site, and Sears (1963) did stratigraphic testing in a few undisturbed areas. The beach component was not sampled by previous studies because it was covered by salt marsh. Coll. and subm. 1965 by D. Phelps.

2962 ± 120

FSU-67. Tucker, Norwood **1012 B.C.**

Charred fiber from sherd interiors of the types Norwood Plain and Norwood Simple Stamped recently described by Phelps (1965). *Comment:* date fits into the reported range of fiber-tempered ceramics in SE U.S. (Bullen, 1961).

1605 ± 325

FSU-66. Tucker, Gulf Check Stamped A.D. 345

Charred organic residue from exterior of vessel of the type Gulf Check Stamped (Willey, 1949). *Comment:* no absolute dates have been previously reported for this type.

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