

Radiocarbon

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BROOKLYN COLLEGE RADIOCARBON DATES I

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INTRODUCTION

This radiocarbon laboratory was established by a grant from Brooklyn College to support the work of its archaeologists and geologists. The method of dating is that of benzene synthesis and liquid-scintillation counting developed by a number of investigators (Noakes *et al*, 1965; Polach and Stipp, 1967; Tamers, 1975). This list includes nearly all samples processed so far, most of which were done since September 1975.

Samples are examined for obvious contamination and are chemically pretreated as noted in each sample description. If necessary, the sample is soaked in warm 1M NaOH to remove humic acids, and rinsed with distilled water until the rinse water is neutral. Then the sample is soaked in warm 1M HCl to remove carbonates and rinsed with distilled water and dried under vacuum in a lyophilizing unit. Carbonate samples are treated as noted.

Samples such as wood, charcoal, and peat, are burned in a stream of oxygen, Linde, extra-dry grade. The combustion products are passed over CuO (at 700°C) and through gas-washing bottles containing KMnO₄ and H₂SiO₄ to remove oxides of nitrogen and sulfur. An oxide of nitrogen, N₂O₃, blue solid, brown gas, frequently seen in the liquid-nitrogen trap, has been virtually eliminated by the scrubbers. The CO₂ is dried in a trap cooled with dry ice in isopropanol, passed through a tube containing P₂O₅ on glass wool and collected in a trap at liquid-nitrogen temperature. If the initial form of the sample is carbonate, CO₂ is produced by the action of a mineral acid and is dried and collected as above. The CO₂ is then allowed to sublime into storage tanks for measurement of the yield. A small volume of CO₂ can be drawn off at this point and reserved for measurements of $\delta^{13}\text{C}$.

Lithium, Li shot, low-sodium, packed under Ar, Lithium Corp America, 25% in excess of the stoichiometric quantity, is placed in a stainless-steel reaction chamber and heated to 600°C, pumping all the time through a trap cooled with dry ice. The temperature is measured with a thermocouple in contact with the bottom of the chamber. The CO₂ is gradually passed through a dry-ice trap onto the Li, not allowing the indicated temperature to exceed 650°C. After all the CO₂ has re-

acted, the temperature is raised to 850° to 900°C and the chamber is continuously evacuated through the dry-ice trap for 30 min to an hour.

After cooling, ca 1L of distilled water is slowly added to the Li_2C_2 . The ensuing C_2H_2 is passed through 2 traps cooled by dry ice and then through a column of P_2O_5 to a liquid-nitrogen trap. The H_2 is pumped off. If fresh Li is used and conditions are properly controlled, yields of 95%, with respect to the CO_2 , may be obtained. The effectiveness of the Li is improved if it is stored under Ar rather than air.

The C_2H_2 is now sublimed directly onto the catalyst, in a tube cooled in a water bath. The entire benzene synthesis including set up and clean up can be done in a day.

Once the C_2H_2 has been absorbed by the catalyst, the tube is sealed by closing a stopcock and can be stored indefinitely. The tube is heated to 150°C for 1 to 2 hr and the C_6H_6 is collected in a dry-ice trap.

The catalyst, developed by Noakes and available from TASK, Inc, Oak Ridge, Tennessee, consists of an alumina base impregnated with vanadium in the +5 oxidation state. 25g of the catalyst, sufficient for the trimerization of the C_2H_2 produced from 12L (STP) of CO_2 , are heated to 350 to 400°C under vacuum through a mechanically refrigerated cold trap at -50°C for a minimum of 4 hr and preferably overnight. The amount of catalyst can be reduced proportionately if the quantity of CO_2 is <12L. The uptake of C_2H_2 by the catalyst, however, will be faster if more catalyst is used.

The results of a brief study of the rate of uptake of C_2H_2 by the catalyst and the percent yield of benzene vs the amount of catalyst are shown in Table 1. The catalyst samples were from the same batch and were all dried at 350°C for 18 hr. The acetylene was produced by the hydrolysis of CaC_2 , Fisher, Electrolite grade, 20 to 30 mesh, using the techniques described above for the Li_2C_2 hydrolysis.

TABLE I
Rate of C_2H_2 uptake and yield of benzene

Catalyst	Time*	Benzene yield	% yield
25g	45 min	5.09g	86
50g	38 min	4.36g	73
100g	20 min	3.57g	60

* Time to absorb 5.5L of C_2H_2 at 1 atm pressure. C_2H_2 pressure kept constant at 1 atm during absorption step.

Benzene for the modern-standard sample is synthesized from CO_2 produced in the wet oxidation of NBS oxalic acid. Hot KMnO_4 solution is run into a solution of the $\text{H}_2\text{C}_2\text{O}_4$ acidified with H_2SO_4 . The CO_2 produced is dried and trapped as described above. The endpoint of the reaction is easily seen by the change from a clear solution to purplish-brown. 10 to 20ml more of the KMnO_4 solution are added, the solution

is stirred for a few min, and then it is allowed to boil briefly under vacuum to remove the last traces of CO₂ from the solution. Analysis, by Teledyne Isotopes, of the CO₂ produced in this method gave a value of $\delta^{13}\text{C}$ equal to -19.44% with respect to PDB.

The benzene is transferred to a counting vial and weighed. If the amount of benzene is $< 4\text{ml}$, the difference is made up by adding benzene, Baker Analyzed Reagent, Spectrophotometric grade; if the amount is greater, an appropriate volume is removed. As can be seen by comparing the results of the counts on 4ml and 0.35ml of benzene synthesized from Sample BC-7, the result does not depend on the amount of benzene produced except that the relative uncertainty is larger for the smaller amount of benzene. Total volume of the solution is made up to $5 \pm 0.05\text{ml}$ by adding 1ml scintillator solution consisting of 50g PPO and 2.5g bis-MSB in 1L toluene.

Counting vials used are commercially available liquid-scintillation vials of low-potassium glass, modified to a volume of 6ml and shielded top and bottom by a disk of old lead 7mm thick. The cork cap liner is replaced by a Teflon disk and the threads are wrapped with Teflon tape. For all vials used, evaporation of benzene is negligible over the counting period, eg, a modern-standard sample in use for 6 mos lost only 30mg . Background in all vials was measured and, relative to each other, all vials exhibit the same background within an average deviation of 3% . A check of 13 vials counted sequentially over a 4 mos period revealed that each vial had a background relative to the average background for all vials by a factor ranging from 0.932 to 1.07. Although for most samples the correction for the relative background is nearly negligible, it is applied for all samples.

All samples are weighed before and after counting to check for possible benzene loss. The samples can be stored for later recount and the modern standard and background, the latter, 4ml Spectrophotometric grade benzene plus 1ml scintillator solution, samples can be used repeatedly, a considerable saving of time, effort, and NBS oxalic acid.

The counter is a Picker Nuclear Liquimat Model 220 with photomultiplier tubes selected for low noise. The samples, including background and modern standard, are placed in the counter and counted sequentially for 100 min/count . The cycle is repeated as often as desired with a minimum of 20 cycles, 2000 min/sample , for each series of determinations. This procedure clearly reveals erroneous 100-min counts and monitors the background continuously during the entire run which may take a week or more.

The constant cycling of the samples has no effect on the respective activities as there is negligible leakage from the vials under the operating conditions. The stability of the system was checked and, as shown in Table 2, was very good.

All samples are checked for quenching by an external ^{137}Cs source and the channels-ratio technique. Quenching is not a problem due to

TABLE 2
Consistency of counter system. 5g benzene solution

Counting period	Background (cpm)	Modern standard $\times 0.95$ (cpm/g benzene)
7/20-7/26/75	2.57 ± 0.05	7.83 ± 0.05
7/27-8/1	2.40 ± 0.04	7.86 ± 0.04
8/2-8/12	2.30 ± 0.04	7.74 ± 0.04
8/13-8/18	2.28 ± 0.05	7.86 ± 0.05
8/19-8/25	2.17 ± 0.05	7.77 ± 0.05
9/12-9/17	2.91 ± 0.05	7.57 ± 0.05
9/18-9/24	2.65 ± 0.05	7.75 ± 0.05

the high purity of the benzene. In an initial period of testing, a modern-standard sample in use for 6 mos, suffered ca 2% decrease of activity. A change in the channels ratio indicated that the problem was quenching; there was no benzene loss. As I had observed that oxygen dissolved in the counting solution would quench the sample, the sample was opened and argon was briefly blown in to displace the air in the vial. This procedure restored the activity and the channels ratio resumed its normal value. Samples are now routinely sealed with an argon atmosphere in the counting vial.

The raw data are scanned for infrequent spurious counts that are discarded. After the respective sample activities are corrected for background and divided by the mass of synthesized benzene, the ratio of the sample activity to that of the modern standard ($\times 0.95$) is calculated.

The fact that the actual measurements in radiocarbon dating produce an activity ratio rather than an age may be easily overlooked. Although ages are often reported in *Radiocarbon* to 4 or 5 significant figures, requiring ca 10^5 and 10^7 counts, respectively, the reported uncertainties for some of these ages indicate that the ratio was not actually determined to that degree of accuracy. In this laboratory, the number of significant digits in the reported age is the same as that determined for the activity ratio, generally 3. The reported uncertainty is a 1σ standard deviation based on counting statistics only.

It would seem reasonable to propose that dating labs should report, in this journal, the actual experimentally determined value for the ratio and uncertainty. This procedure would be advantageous in that while the calculated ages as reported are based on a number of assumptions, such as choice of half life and dendrochronologic correction, the ratio has enduring significance and is the basic datum to which one must return if the procedures of calculating age change.

Where desired by the submitter, the age is calculated using the 5730 yr half-life and corrected using the MASCA table (Ralph *et al*, 1973). Table 3 shows the results of some cross checks.

TABLE 3
Results of cross checks

Sample	Measured age	Our measurement	MASCA correction
	Years BP, $t_{1/2} = 5568$		
Charcoal, (U Ga)	3455 ± 85	3880 ± 100	—
Charcoal, (U Ga)	540 ± 55	490 ± 90	—
Peat, (SI-1103)	11,100 ± 120	9500 ± 400	—
Sequoia wood, P-SW-SEQ-2	1850 — 1860	1980 ± 60	1930 ± 90
(6 samples, 10 rings each.	1708 — 1718	1570 ± 60	1550 ± 80
Age dendrochronologically	1466 — 1476	1680 ± 60	1670 ± 100
determined, see Ralph <i>et al</i>)	1331 — 1341	1250 ± 60	1150-1250 ± 70
	1200 — 1210	1210 ± 60	1190-1160 ± 60
	1040 — 1050	910 ± 70	900 ± 90

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SAMPLE DESCRIPTIONS

I. GEOLOGIC SAMPLES

BC-20. Piston Core E-30-71**4300 ± 70****2380 BC**

Shell (*Mulinia* sp and *Nucula* sp) from core sample Delaware Bay, (39° 5' 20" N, 74° 13' 70" W). Water depth 9.4m, referred to mean low water, depth in core 2.2m. Sediment matrix was a very fine sandy mud. Coll 1971 by C B Weil, Dept Geol, Brooklyn Coll. *Comment* (CBW): this core was taken during a study of transgressive sedimentary environments of Delaware Bay. The date falls on Kraft's (1971) sea-level-rise curve for the Delaware coastline and indicates rate of local sediment deposition.

Cave series

Deposits on cave walls and enlarged joints consisting of a resinous solid containing visible fecal matter and plant fragments. Coll by David Leveson, Dept Geol, Brooklyn Coll. NaOH/HCl pretreatment used.

BC-21. Canyonland**2900 ± 120****950 BC**

Coll April, 1972 from wall of cave formed by joint enlargement, Cutler-Rico formation (Permian) Needles Dist, Canyonland Natl Park, Utah (38° 6' N, 104° 52' W).

BC-22. Zion**3840 ± 50****1890 BC**

Coll April, 1975 from rear of "Pictograph" Cave, Navajo Sandstone, Rock Pasture area of Zion Natl Park (37° 10' N, 112° 53' W).

Coles Cave series

Pleistocene stalactites precipitated on roof of Coles Cave by vadose seepage, Barbados, West Indies (13° 10' N, 59° 30' W). Coll 1966 by W H Harris, Dept Geol, Brooklyn Coll. Samples were growth-layer increments of conical ring stalactites composed of low-magnesium calcite; dimensions refer to distance from original outer surface of stalactite. $\delta^{13}\text{C}$ referred to PDB.

BC-23. Stalactite 3, Layer S1-C**7740 ± 140****5790 BC** $\delta^{13}\text{C} = -9.66 \pm 0.2\text{‰}$

9 to 17.5mm. With MASCA correction and zero-age correction (see comment at end of series) age is 3500 \pm 1700
– 1200

BC-24. Stalactite 3, Layer S2-T1**5920 ± 90****3970 BC** $\delta^{13}\text{C} = -9.66 \pm 0.2\text{‰}$

2.5 to 9mm. MASCA and zero-age corrected date: 1300 \pm 970
– 700

BC-25. Stalactite 4, Layer S1-3**10,100 ± 140****8150 BC** $\delta^{13}\text{C} = -8.8 \pm 0.2\text{‰}$

14.8 to 39.5mm. MASCA and zero-age corrected date: 6600 \pm 1700
– 1300

BC-26. Stalactite 4, Layer S2-4**9800 ± 130****7850 BC** $\delta^{13}\text{C} = -8.8 \pm 0.2\text{‰}$

8.5 to 36mm. MASCA and zero-age corrected date: 6400 \pm 1700
– 1100

BC-27. Stalactite 4, Layer S3-4A**9760 ± 400****7810 BC** $\delta^{13}\text{C} = -8.8 \pm 0.1\text{‰}$

4 to 25mm. MASCA and zero-age corrected date: 6360 \pm 2000
– 1500

General Comment (WHH): by extrapolation of dates of growth rings, a zero-age for stalactites was obtained. This “zero-age”, 4700^{+840}_{-630} , reflects admixture of fossil carbon that is ^{14}C inactive via limestone dissolution in the vadose zone and bulk atmospheric carbon from when the seepage left the soil zone. If ages are compared with conventional calendar scales, 4700 yr age correction must be subtracted. Corrected dates listed for samples are based on 5730 yr half-life.

Marie-Galante Coral series

Aragonite coral samples from elev reef terraces on Marie-Galante I., Guadeloupe. The lowest terrace, at +1 to 3m, is constructional and can serve as a marker of a deglacial event (rising sea level) in the Pleistocene. Coll July, 1974 by N E Pingitore, Jr, Dept Geol, Brooklyn Coll.

BC-28. Coral MG-10 **30,300 ± 600**
28,300 BC

Surface coral sample, from reef elev 3m, reef core, in growth position, Anse Talisronde, E coast, Marie-Galante (15° 56' N, 61° 12' W).

BC-29. Coral MG-14 **35,000 ± 2,000**
33,000 BC

Surface coral sample, reef elev 3m, from backreef lagoon sediments. S coast, due S of Thibault (15° 52' N, 61° 16' W).

BC-30. Coral MG-17 **31,400 ± 800**
29,400 BC

Surface coral sample, 2nd terrace of I. elev ca 15m, S coast, Marie-Galante, 1km E of airport (15° 52' N, 61° 16' W).

General Comment (NEP): contamination by more recent carbon may have occurred, but aragonite mineralogy, determined by x-ray diffraction, casts doubts on possibility. If dates are real, they indicate a warming trend (deglacial event) at ca 30,000 BC. Uranium-helium measurements will be made to confirm ages.

BC-39. Caliche—14-16 **22,000 ± 1000**
20,000 BC

Caliche from Odessa, Texas in Permian Basin (31° 52' N, 102° 23' E). Stratum, overlying Pleistocene lake deposits, dated at 4000 to 8000 BP, based on stratigraphic evidence. Coll March, 1976 by S J Mazzullo, Univ Texas, Permian basin; subm by J M Mazzullo, Brooklyn Coll. *Comment* (SJM): date indicates reworking of caliche.

II. ARCHAEOLOGIC SAMPLES

A. Europe

Susani series

Late Bronze Age samples from the Susani site Lugoj, Timis region, Comm Traian, Vulia, Romania (46° 20' N, 39° 32' E) (Popescu 1969). Coll 1968 by I Straton, Lugoj Mus, Lugoj, Romania; subm by H A

Bankoff, Dept Anthropol, Brooklyn Coll. All samples found in a “sanctuary”, a round tumulus-like structure of broken earth, with several pits containing burnt organic matter and charred grain. Selected by the excavator, I Staton, to establish chronology of pits and burnt levels. *Comment*: NaOH/HCl pretreatment.

BC-1. Burnt—Level 1, Meter 10 **3110 ± 70**
1160 BC
MASCA corrected date: 1460-1480 $\begin{matrix} + 60 \\ - 120 \end{matrix}$ BC

Charcoal from Meter 10 within Burnt-Level 1, assoc with Hallstatt A/B pottery. Date, using 5730 t $\frac{1}{2}$: 1250 ± 70 BC.

BC-3. Offering Pit 1 **3450 ± 60**
1500 BC
MASCA corrected date: 1800 $\begin{matrix} + 240 \\ - 80 \end{matrix}$ BC

Carbonized grain within Offering Pit 1, under Burnt-Level 1, assoc with >100 single-handled cups of typical Early Hallstatt type. *Comment* (HAB): probably Hallstatt A, similar to cups from Bobda, Romania. Date, using 5730 t $\frac{1}{2}$: 1600 ± 60 BC.

BC-4. Burnt—Level 1, Meter 17-18 **3330 ± 80**
1380 BC
MASCA corrected date: 1690 ± 90 BC

Burnt wood, Burnt-Level 1, Meter 17-18. Date, using 5730 t $\frac{1}{2}$: 1470 ± 80 BC.

General Comment (HAB): dates are ca 500 yr earlier than expected, if MASCA correction is applied. Uncorrected dates, using 5730 yr half-life, fit accepted chronology better.

BC-2. Omoljica **3530 ± 80**
1580 BC
MASCA corrected date: 2050 ± 70 BC

Carbonized grain. Coll by V Karapandzic, Narodni Muz Beograd; subm by H A Bankoff. Originally coll at Omoljica, Banat, Yugoslavia (44° 53' N, 20° 40' E). Sample was found in storeroom of Narodni Muz in a typical 2-handled Pancevo-Omoljica type “beaker” of Middle Bronze (B-C) age date. Inventory No: 344-345. Tested as check for dates from excavated context from Mediana. Date, using 5730 t $\frac{1}{2}$: 1690 ± 80 BC (Trbuhovic, 1967). *Comment*: NaOH/HCl pre-treatment.

Mediana series

Samples from Late Bronze age site of Brzi Brod, Mediana, near Nis, Yugoslavia (43° 20' N, 21° 50' E) (Garasanin, 1969). Coll Aug, 1972 by M V Garasanin, Univ Beograd; subm by H A Bankoff. *Comment*: NaOH/HCl pretreatment.

BC-5. Pit 1, Lj XVII **3530 ± 60**
1580 BC
MASCA corrected date: 2050 ± 70 BC

Carbonized wood from center of Pit 1, Lj XVII, 1st level. Date, using 5730 t½: 1690 ± 60 BC. *Comment* (HAB): agrees well with BC-2, supporting Garasanin's placement for lowest level of Mediana in Middle Bronze age (Bronze C?). Other dates in this series do not agree.

BC-6. Pit 1, Lj VIII **3230 ± 90**
1280 BC
MASCA corrected date: 1570-1600 ± 80

Ashes and carbon flakes from area in front of LBA construction, Lj, VIII, Pit 1, No. 65 E 4.00. Date, using 5730 t½: 1370 ± 90 BC.

BC-7. Pit 2, Lj XVIII **3940 ± 40**
1990 BC
MASCA corrected date: 2560 ⁺³⁰/₋₇₀ BC

Wood (oak) from bottom of Pit 2, Lj XVIII. Date, using 5730 t½: 2110 ± 40 BC. *Comment:* sample, comprised of 4ml synthesized benzene, + 1ml scintillator solution. A 2nd sample was made from 0.3ml synthesized benzene, left over after 4ml was taken, + 3.7ml inert benzene and counted. Age of BC-7, determined using latter sample, was 3800 ± 200 BP agreeing well with above.

BC-8. Pit 1, Lj XVII **3000 ± 200**
1000 BC
MASCA corrected date: 1300 ⁺²⁰⁰/₋₃₀₀ BC

Charcoal, center of Pit 1, Lj XVII. Date, using 5730 t½: 1100 ± 200 BC.

BC-9. Lj XVI **3650 ± 80**
1700 BC
MASCA corrected date: 2120-2140 ± 40 BC

Oak, pit under the object, Lj XVI. Date, using 5730 t½: 1800 ± 80 BC.

BC-10. Pit 1, Lj XVIII **3260 ± 100**
1310 BC
MASCA corrected date: 1600-1640 ± 100

Charred wood from interior of Pit 1, Lj XVIII. Date, using 5730 t½: 1410 ± 100 BC.

General Comment (HAB): MASCA-corrected dates run several centuries earlier than chronology based on considerations such as ceramic typology.

BC-42. Log Boat **250 ± 100**
AD 1700

Wood. Hollowed log boat from Sava R bed at depth 4m. Coll by Edward Ochsenchlagler during investigation of Roman site; Sirmium, near Sremska Mitrovica, Yugoslavia (44° 55' N, 19° 35' E). *Comment:*

NaOH/HCl pretreatment. *Comment* (EO): date rules out Neolithic or Roman origins.

B. Egypt

Head rest series

Two wooden headrests in Egyptian coll of Brooklyn Mus. Subm by Robert Bianchi, Brooklyn Mus for authentication and confirmation of age based on stylistic analysis. Samples drilled from bottom.

BC-40. BM No. 05.325 **4000 ± 70**
2050 BC
MASCA corrected date: 2600 ⁺²⁰⁰₋₈₀ BC

Wood headrest from Egypt, context unknown. Date, based on 5730 t_{1/2}: 2160 ± 70 BC. *Comment* (RB): either of dates based on 5730 t_{1/2} and MASCA correction agree with our date based on stylistic analysis which places object in Old Kingdom, 2686 to 2181 BC.

BC-41. BM No. 37.443E **2710 ± 80**
760 BC
MASCA corrected date: 900 ± 110 BC

Wood headrest from Egypt from coll of Henry Abbott, known to be from Saqqara, context unknown. Date, based on 5730 t_{1/2}: 830 ± 80 BC. *Comment* (RB): date, based on stylistic analysis, is 1200 to 1100 BC.

C. United States

Old Fort series

Samples from a site investigated to find evidence for an old fort built by Esopus Indians and later razed by the Dutch in retaliation for a massacre, Ulster County, NY, (41° 37' 30" N. 74° 15' W.) Coll, Aug, 1972 by H D Winters and I A Berman, New York Univ. Charcoal from layer of yellow sand above culturally sterile layer, presuming earliest occupation of site.

BC-35. Feature 17a **4200 ± 100**
2300 BC

Charcoal, 56cm below surface, Feature 17a, hearth. *Comment:* no pretreatment.

BC-36. Feature 17a **4130 ± 90**
2180 BC

Charcoal, same as BC-35. *Comment:* NaOH/HCl pretreatment. Good agreement with BC-35.

BC-38. Feature 7a **3980 ± 50**
2030 BC

Charcoal, Feature 7a, hearth pit. *Comment:* NaOH/HCl pretreatment.

General Comment (IAB): No evidence found for fort; based on typological comparisons, projectile-point types, etc and above dates, site is probably "Archaic."

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