

BRITISH MUSEUM NATURAL RADIOCARBON MEASUREMENTS I

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The first series of radiocarbon dating measurements made at the British Museum Research Laboratory are reported in the following list. The report covers a period during which two instances of radioactive contamination occurred to interrupt dating work, and is consequently a short one.

INTRODUCTION

All measurements were made in the same small mild steel proportional counter (effective volume just under 500 cc) designed by W. R. Loosemore of the Atomic Energy Research Establishment, Harwell. This operates with an acetylene filling at 140-cm Hg pressure and has an integral mercury shield. Acetylene is synthesized by a method described by Barker (1953) and since improved to give consistent yields around 96% with no detectable isotopic fractionation. Gas purity is checked by observing the counter characteristic using a cobalt 60 source at a fixed distance, and the working point is adjusted to compensate for any slight variations in gas purity. The counter is screened by 8 in. of mild steel and 17 geiger counters in anti-coincidence as well as its integral mercury screen. A layer of paraffin wax containing 15% by weight of boric acid is also incorporated in the steel shield to reduce the neutron effect (de Vries, 1957). The "modern" count is 11.3 counts/min and the background 4.3 counts/min at 760-mm barometric pressure. The barometric coefficient of the background is slightly less than 1% cm Hg.

Additional safeguards against inaccuracies include continuous monitoring of the neutron flux in the steel screen as suggested by de Vries, thorough daily checking of the electronic equipment, and the use of a system of mechanical registers which provides a record of the count distribution in the output channels during each period of measurement (Crathorn and Loosemore, 1954). Any departure from normal statistical scatter during an overnight or weekend count is thus quickly detected.

The "modern" reference sample is taken from the 100th tree ring counting inwards from the bark of an oak tree recently felled. This sample is the same as that used by the Cambridge laboratory, and its use ensures that the contemporary value in the age calculation is free from the effect of dilution of the atmosphere with inactive carbon by the large-scale combustion of fossil fuels since the Industrial Revolution (Suess, 1955).

Background measurements are made using acetylene prepared from anthracite. The early work showed that the background remained very steady within the expected statistical fluctuations over a period of many months, apart from the slight barometric fluctuations. Nevertheless, frequent measurements of background and contemporaneous samples are made, for on two occasions during 1958 a rise in background of a few counts per minute was detected. In both cases, the increase of activity was of a temporary nature, and the rate of decay suggested the presence of fission products. It may be significant that each

of these increases in background occurred soon after the announcement of a series of nuclear weapon tests, one in the spring, the other in the autumn, and together they explain why this list is such a short one, since dating measurements were interrupted for a considerable time on each occasion. The occurrences were very puzzling in that the balance of evidence in each case pointed to the sudden appearance of the activity inside the counter under conditions which seemed to make this an impossibility. However, investigations have since shown that air-borne activity may have entered through an intermittent leak in a vacuum union, and steps have been taken to prevent a recurrence of the trouble.

All samples have been measured on at least two occasions, with an interval of at least one month between them, in order to demonstrate that there has been no radon contamination. In fact, the method of acetylene synthesis and purification was designed to eliminate radon from the sample, and these measurements have confirmed that this is so.

Correction for isotopic fractionation effects is not possible on a routine basis owing to the lack of suitable mass spectrometric facilities. Because of this, and of the possibility of errors due to the effects recently discovered by de Vries (1958), it is considered unrealistic to quote errors based solely on the counting statistics. Error terms have been widened therefore to include contributions of ± 80 yr for isotopic fractionation effects, and ± 100 yr for the de Vries effects. Ages are calculated on a half-life of 5568 ± 30 yr.

Pretreatment of samples.—All organic material is treated with dilute 1% hydrochloric acid in order to eliminate the possibility of contamination with carbonates. Charcoal samples are extracted thoroughly, first with hot 1% hydrochloric acid and then with hot 1% caustic soda. This should remove both carbonate and humic contamination. When the treatment is complete, the charcoal is washed with 1% hydrochloric acid to free it from alkali and finally with distilled water to remove the acid.

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SAMPLE DESCRIPTIONS

I. CHECK SAMPLES AND SAMPLES OF KNOWN AGE

BM-16. St. Cuthbert, Durham, England 1333 \pm 150

Wood from the original coffin of St. Cuthbert, Durham Cathedral ($54^{\circ} 47' N$ Lat, $1^{\circ} 34' W$ Long). Sample made available by permission of the Dean and Chapter of Durham Cathedral. There are a number of coffins associated with the bones of the saint, but the original inner one almost certainly dates to A.D. 698. The known age of the sample is thus 1270 yr plus the age of the timber. The agreement with the radiocarbon age is satisfactory.

able by C. Cortesi and M. Beneventano, Carbon-14 laboratory, Rome. Known age 1908 yr plus the age of the timber. Dated by the Stockholm laboratory (St-103A.B, 2010 ± 65 ; Östlund, 1957), by the Cambridge laboratory (Q-112, 1904 ± 95 ; Godwin and Willis, 1959), and by the Uppsala laboratory (U-68, 1980 ± 70 ; Olsson, 1959).

BM-22. Sesostris, Egypt **3530 \pm 150**

Wood from the deck of the funerary ship of Sesostris III. Originally subm. to the Chicago laboratory by Colonel C. C. Gregg, Chicago Natural History Museum, and made available to the British Museum laboratory by W. F. Libby. Known age 3750 yr, according to John Wilson, Oriental Institute, University of Chicago. Dated by the Chicago laboratory (C-81, 3621 ± 180 ; Arnold and Libby, 1951).

BM-18. Tree Ring Sample, Sequoia **2803 \pm 150**

Wood from the heart of the giant redwood known as Centennial stump, felled in 1874, with 2905 rings between the innermost (and 2802 rings between the outermost) portion of the sample and the outside of the tree. Therefore, the known mean age was 2928 ± 51 yr. Sample subm. to the Chicago laboratory by E. Schulman, Laboratory of Tree Ring Research, University of Arizona. Made available to the British Museum by W. F. Libby. Dated by the Chicago laboratory (C-159, 2710 ± 130 ; Arnold and Libby, 1951).

BM-19. Ruds Vedby **11,333 \pm 200**

Wood from the zone boundary between the Allerød and the younger Dryas periods, Ruds Vedby ($55^{\circ} 34' N$ Lat, $11^{\circ} 25' E$ Long), Zealand, Denmark. Sample made available by H. Tauber of the Copenhagen laboratory where it was dated by the solid-carbon method (K-101, $10,890 \pm 240$; Anderson, Levi, and Tauber, 1953); also dated by the Uppsala laboratory (U-20, $10,830 \pm 130$; U-75, $10,680 \pm 130$; Olsson, 1959) and by the Stockholm laboratory (St-18, $10,200 \pm 300$; Östlund, 1957).

II. ARCHAEOLOGIC SAMPLES

A. Africa

Holley Shelter series, Wartburg

Two samples of charcoal from the excavations in progress in a rock shelter on Messrs. Holley Bros. farm "Everdon" at Wartburg ($29^{\circ} 26' S$ Lat, $30^{\circ} 35' E$ Long), Natal, South Africa. The site is described by Cramb (1950). BM-30 is from the 24- to 30-in. layer of grid Z 3 and was collected from an ancient hearth together with bone fragments and Middle Stone Age implements. BM-34 is from the 18- to 24-in. layer of grid Z 2 which adjoins grid Z 3. The grids are 3 ft square, so that the samples are quite close to each other, both horizontally and vertically. The Middle Stone Age material is homogeneous throughout. Bantu and Later Stone Age material is rarely more than 6 in. below the surface. Coll. by Gordon Cramb, of 5 Old Mill Way, Durban North; subm. by the Director, Archaeological Survey, Union of South Africa.

BM-30. Holley Shelter I **18,200 \pm 500**
Charcoal from the 24- to 30-in. layer of grid Z 3.

BM-34. Holley Shelter II**4490 ± 150**

Charcoal from the 18- to 24-in. layer of grid Z 2. *Comment:* the close proximity of the samples makes the difference in their ages seem extraordinarily high. However, the excavator has pointed out that the situation of the cave is such that the build-up of the floor level may have been extremely slow. The value for BM-30 is not inconsistent with another Middle Stone Age date (C-925 Cave of Hearths, 15,000 ± 730; Libby, 1956). On the other hand, BM-34 is much too young by comparison with other Middle Stone Age dates, and one must assume either that the sample is suspect or that the Middle Stone Age people lived uninterruptedly in these parts to a period more recent than archaeological work elsewhere in South Africa indicated. Both samples were given the standard pretreatment for charcoal.

BM-39. Olieboompoort Cave, Waterberg**>25,000**

The cave is on the west side of Olieboompoort, NW of Waterberg, Transvaal (23° 50' S Lat, 27° 40' E Long) between Ellisras and Vaalwater villages (see South Africa 1:500,000 Topographic sheet Pietersburg SE 25/26). Sample of heavily mineralized material resembling charcoal but probably decayed wood from Bed 2. The nodules were scattered between 36 to 54 in. below the present-day cave floor, in a rich Middle Stone Age Pietersburg culture horizon, i.e. they do not appear to represent a fireplace. Coll. September-October, 1954 by Dr. R. J. Mason during excavations financed by the Wenner-Gren Foundation; subm. by the Director, Archaeological Survey, Union of South Africa. Relevant publications—Mason (1957, 1958). *Comment:* The sample is from a Pietersburg culture "Middle" Stone Age horizon and is, therefore, earlier than the later stage of the Pietersburg culture which, in the case of the Cave of Hearths, has a date of 15,100 ± 730 (C-925). An earlier date than 15,100 ± 730 B.P. would, therefore, be expected, but >25,000 seems to be too early if the Cave of Hearths date is correct. The sample closely resembled charcoal, but nothing remained after treatment with hot 1% hydrochloric acid and hot 1% caustic soda. Accordingly, the caustic-soda-soluble fraction was reprecipitated with acid, washed and dried, and used as the source of carbon. Under these circumstances one cannot rule out the possibility of contamination. There are also inconsistencies in the Cave of Hearths dates (C-924 to C-927, Libby, 1956), and the problem will remain unresolved until further South African material is dated.

*B. Central America***BM-37. Las Cuevas, British Honduras****920 ± 150**

Sample of charcoal from the brown earth layer in trench 7 (in front of floor Z) of the outer cave at Las Cuevas, approx. 35 mi S of Cayo (17° 14' N Lat, 89° 03' W Long), British Honduras. The sample was in association with sherds suggesting the late Classic period, and with three-pronged incensaries. Coll. February 1957 by Adrian Digby, Keeper of Ethnography, British Museum. Relevant publication—Digby (1958). *Comment:* the sample was given the standard pretreatment for charcoal.

C. Egypt

BM-21. Mentuhotep, Thebes **3580 ± 150**

Section of tree trunk from the mortuary temple of Neb-hepet-Re Mentuhotep (XIth Dynasty) at Deir el Bahri, Thebes (25° 44' N Lat, 32° 38' E Long), Egypt. Found by the Egypt Exploration Fund 1907 and now in the Department of Egyptian Antiquities, British Museum No. 47791. Expected age 2010 B.C. (3968 B.P.) is based on astronomical evidence and should not be more than 20 yr in error. *Comment*: the age obtained is significantly younger than expected.

BM-27. Hemaka, Saqqara **4100 ± 150**

Wood, id. by the Forest Products Research Laboratory as a species of *Acacia*, probably *A. arabica*. From a brick mastaba at Saqqara (29° 50' N Lat, 31° 12' E Long), Egypt. The tomb was that of a nobleman, Hemaka, and dates to the reign of Udimu (Ist Dynasty). Coll. 1937 by W. B. Emery; subm. by the Department of Egyptian Antiquities, British Museum. Relevant publication—Emery (1938). Expected age 4958 ± 200. *Comment*: the discrepancy is large. Sample dated by the Pennsylvania laboratory (P-214, 4447 ± 150; Ralph, 1959).

D. Great Britain

BM-17. Hod Hill, Dorset, England **1570 ± 150**

Charcoal from the Iron Age "AB" occupation layer associated with the first stage of the main Iron Age rampart (50° 53' 40" N Lat, 2° 12' W Long), 3.25 mi NW of Blandford, Dorset. Coll. 1957 and subm. by J. W. Brailsford, Department of British and Mediaeval Antiquities, British Museum. *Comment*: the age is some centuries younger than the latest accepted archaeological dating.

BM-40. High Rocks Shelks, Tunbridge Wells, England **5660 ± 150**

Sample of charcoal from an undisturbed hearth (No. 5) in site F (51° 7' 20" N Lat, 0° 13' 40" E Long). Associated with Mesolithic sherds. Stratigraphically it belongs to the later Mesolithic phase at High Rocks. Coll. August, 1956 and subm. by J. H. Money, F.S.A. Publication due in the 1959 volume of the Sussex Archaeological Collections. *Comment*: the sample was given the standard pretreatment for charcoal.

III. GEOLOGIC SAMPLE

BM-29. Mounts Bay, Cornwall, England **3656 ± 150**

Oak from a submerged forest (50° 03' N Lat, 5° 25' W Long). Coll. from below the normal low-tide mark opposite Larrigan, between Penzance and Newlyn. Date of collection believed to be 1883. Subm. by Sir Gavin de Beer, Director, British Museum (Natural History). Photographs of the trees appear in the Transactions of the Royal Cornish Geological Society, 1914, v. 13, p. 312, and reference is made to them on p. 313-318. *Comment*: there is an ancient tradition that St. Michaels Mount was at one time situated in a wood, and these trees must have formed part of it. Their growth would have been

stopped by the changes which led to the Mount becoming an island in the Bay. Pretreatment consisted of extraction with hot 1% hydrochloric acid followed by washing with distilled water.

REFERENCES

- Anderson, E. C., Levi, Hilde, and Tauber, Henrik, 1953, Copenhagen natural radiocarbon measurements, I: *Science*, v. 118, p. 6-9.
- Arnold, J. R., and Libby, W. F., 1951, Radiocarbon dates: *Science*, v. 113, p. 111-120.
- Barker, H., 1953, Radiocarbon dating: Large-scale preparation of acetylene from organic material: *Nature*, v. 177, p. 631-632.
- Cramb, G., 1950, A Middle Stone Age industry from a Natal rock shelter: *South African Jour. Sci.*, v. 48, p. 181-186.
- Crathorn, A. R., and Loosemore, W. R., 1954, Gas counting of natural radiocarbon: *Radioisotope Conf.*, 1954, v. 2, p. 123-131. [London, Butterworth's Scientific Pubs.]
- Digby, A., 1958, A new Maya city discovered in British Honduras: *Illustrated London News*, v. 232, p. 274.
- Emery, W. B., 1938, The tomb of Hemaka: Cairo, Egyptian Govt. Service des Antiquités.
- Godwin, H., and Willis, E. H., Cambridge University natural radiocarbon measurements I: *AM. JOUR. SCI. RADIOC. SUPP.*, v. 1, p. 63-75.
- Libby, W. F., 1955, Radiocarbon dating, 2d ed.: Chicago, Chicago University Press, 175 p.
- Mason, R. J., 1957, The Transvaal Middle Stone Age and statistical analysis: *South African Archaeol. Bull.*, v. 12, p. 119-143.
- 1958, Bone tools at the Kalkbank Middle Stone Age site: *South African Archaeol. Bull.*, v. 13, p. 85-88.
- Olsson, Ingrid, 1959, Uppsala natural radiocarbon measurements I: *AM. JOUR. SCI. RADIOC. SUPP.*, v. 1, p. 87-102.
- Östlund, H. G., 1957, Stockholm natural radiocarbon measurements I: *Science*, v. 126, p. 493-497.
- Ralph, E. K., 1959, University of Pennsylvania radiocarbon dates III: *AM. JOUR. SCI. RADIOC. SUPP.*, v. 1, p. 45-58.
- Suess, H. E., 1955, Radiocarbon concentration in modern wood: *Science*, v. 122, p. 415-417.
- Vries, Hl. de, 1957, Further analysis of the neutron component of the background of counters used for ^{14}C age measurements: *Nuclear Physics*, v. 3, p. 65-68.
- 1958, Variation in concentration of radiocarbon with time and location on Earth: *Koninkl. Nederlandse Akad. Wetensch. Proc.*, ser. B, v. 61, no. 2, p. 1-9.