CHAPTER 13

RADIOCARBON DATE CALIBRATION USING HISTORICALLY DATED SPECIMENS FROM EGYPT AND NEW RADIOCARBON DETERMINATIONS FOR EL-AMARNA

by

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13.1 Egyptian historical chronologies

From the inception of the radiocarbon dating method there has been much interest in the dating of samples from ancient Egypt. No other ancient civilisation has left a legacy of such great quantity and variety of datable materials spanning so long a range of time. Using disparate methods, it is generally possible to assign dates to many Egyptian events and objects and hence construct a comprehensive historical chronology. Despite inevitable scholarly disagreements over detail, the main chronology appears to be a rigid structure of interlocking facts. Nevertheless there is always the remote possibility of bias.

A foundation stone of this edifice is one of the three calendars used by the ancient Egyptians, the Civil Calendar. It had evidently been devised to begin with the onset of the annual Nile inundation, and more particularly with an astronomical event which occurred at this time of year: the first day that the star Sirius appeared on the eastern horizon immediately prior to the rising sun (the heliacal rising of Sirius). In the absence of a leap year in this calendar, however, the first day advanced by one day in four years with respect to the solar year, resulting in the coincidence of the heliacal rising of Sirius with the first day of the calendar year only once in about 1460 years. This is known as the Sothic Cycle and varies slightly. From surviving documents and astronomical data it has been possible to fix three of the heliacal risings of Sirius (Parker 1950), the earliest verifiable of which was in the 7th year of the reign of Sesostris III of the Twelfth Dynasty and occurred in 1872 B.C. These dates are the basis on which the chronology of ancient Egypt has been bnilt using king lists and other documents for interpolation.

13.2 Establishing radiocarbon dating

Because of this well-fonnded time scale, W.F. Libby chose to use specimens of known age from Egypt to test his newly proposed dating method. With the aid of a small committee of the American Anthropological Society and the

Geological Society of America he obtained museum specimens that were well authenticated and dated by these specialists. Amongst other specimens there were four pieces of timber from Egypt from a period extending from 200 B.C. to 2650 B.C. The radiocarbon content of these was measured and the results were shown to be consistent with the radiocarbon decay curve using a value of 5720 ±47 years for the half-life of the 14C isotope. This value had been obtained in Libby's earlier work on the project and was the best value then available. The results "seemed sufficiently encouraging to warrant further investigation of the method" (Libby and Arnold 1949). Compared to present-day measurements these appear rather crude since the precisions obtained ranged from about 5% to 19% for samples less than one half-life in age. With modern equipment the expectation would be better than 1%, and the latest calibration curves are made with precisions of about 0.2% to 0.3%, which represent about ±17 years. The method found widespread acceptance and increasing numbers of dating laboratories are still being established. During the following decade many samples from ancient Egypt were dated. Progress in techniques was then rapid, but as precision of the results increased discrepancies began to emerge between dates obtained from the specimens and the dates predicted by the specialists.

13.3 Discrepancies

Papers dealing with discrepancies appeared but it was difficult to decide whether the anomalies were inherent in the samples themselves, were the result of uncertainties in the chronology, especially for the earlier periods (e.g. Smith 1964; Mellaart 1979), were inherent in the radiocarbon method, or whether the cause was that the radiocarbon techniques in use were still inadequate. Libby (1963) discussed the accuracy of radiocarbon dating and suggested possible explanations for the deviations. One of these was the possible variation of the radiocarbon content of the atmosphere at different times. A way of investigating this was through the record preserved in the annual growth-rings of trees in temperate climates. De Vries had noted an apparent excess of radiocarbon in trees growing during the period of the Little Ice Age. The first systematic study of the phenomenon was initiated at the Cambridge radiocarbon dating laboratory (Willis, et al. 1960), where a *Sequoia gigantea* provided samples of wood of accurately known age. The radiocarbon content of these rings was measured at three collaborating laboratories in an attempt to avoid bias, and it demonstrated that there were indeed variations of the order of 1% during the past 1800 years.The significance of this was immediately apparent to the radiocarbon dating community, since the dating method has as one of its basic tenets the constancy of radiocarbon in the atmosphere over extended periods. It was important to learn the extent of this variation and how long it persisted. Several projects on tree-ring measurements were established in different parts of the world including Germany, Canada, Japan, Argentina and the U.S.A. The results of these tests were sufficient to initiate a research effort attempting to quantify the atmospheric variations. This has since involved many laboratories for more than a quarter of a century. Most of the specimens used in this research were initially provided by the Laboratory for Tree-Ring

Research, Tucson, Arizona, which institution ensured that they were accurately dated by dendrochronological techniques (Ferguson 1970). The main samples were from the Giant Redwood, Ponderosa Pine and the Bristlecone Pine, and were supplied principally to the radiocarbon laboratories at La Jolla, Philadelphia and Tucson. The extent of this work may be gauged from the fact that Professor Suess, at La Jolla, has measured some 1000 samples (approximately double the number of those of the other laboratories) which may be used to produce a calibration curve to convert radiocarbon dates to the historical time-scale. Two symposia were held during 1969, in Uppsala and London, in order to discuss the atmospheric variations and their implications.

13.4 Egyptian specimens

The vital importance of the precise provenance of the samples to be dated was impressed upon the archaeologists (Säve-Söderbergh 1970) and projects were set up to procure good specimens with well-established credentials and ages that could be relied upon. Collaboration was arranged between the British Museum and University of California, Los Angeles, and between Uppsala and Philadelphia. With the co-operation of the Egyptian authorities expeditions were arranged to select the most suitable specimens for dating. The results of these studies have now been published (Edwards 1970; Berger 1970; Säve-Söderbergh and Olsson 1970; Michael and Ralph 1970; Olsson and El-Daousay 1979).

13.5 Calibration

During the first half of the 1970's, a series of calibration curves or charts was published. Many authors were involved, but inevitably they were all based principally on the published results of the laboratories at La Jolla, Philadelphia and Tucson. The results required treatment in order to provide an easily readable and usable curve or tables. The treatments all involved some form of averaging or smoothing of the data in order to reduce the scatter apparent amongst the measured points. For final presentation, either curve-fitting routines were applied, or running means calculated. It has been shown that the statistical basis of most of them was not sound, and the uncertainties were not treated properly, if at all. The amount of data now available renders all of these obsolete and they are not now recommended for calibration. The only calibration from that era that may still merit consideration is that of Clark (1975), even though the number of data points considered is much smaller than in the new calibrations recently published.

The radiocarbon dates of the historically dated Egyptian samples were converted to calendar dates using the Bristlecone Pine calibration then available and the results analysed (McKerrell 1975; Clark and Renfrew 1973). The analyses produced conflicting results as to the validity of the Bristlecone Pine calibration of radiocarbon dates. A more recent and complete analysis of the procedures used by these authors (Clark 1978) indicates that the calibrated time-scale is not incompatible with Egyptian historical chronology in the period between 3000 and 300 B.C. These findings will be tested with the new radiocarbon measurements presented here.

13.6 Recent calibrations

With the demise of the radiocarbon dating facility at La Jolla, the flow of measurements on dendrochronologically dated Bristlecone Pine has come to a halt, though a few still remain to be published. H.E. Suess has presented (1979) his definitive calibration table on results obtained up to August 1978, which allows the calibration within the period back to 7000 B.P. (6000 B.C. in calendar years): it is based on the measurements of 1000 samples made entirely at La Jolla.

A workshop devoted to "Calibrating the Radiocarbon Time Scale" was held at Tucson in 1979 to attempt to produce, by a consensus amongst the laboratories, the best statistically derived calibration from 1154 radiocarbon measurements principally made on Bristlecone Pine and Giant Redwood from the laboratories at Tucson, Groningen, La Jolla, Yale and Philadelphia. Comprehensive tables and graphs have since been published (Klein, et al. 1982) allowing the calibration of samples ranging in age from 10 to 7240 B.P., or in calendar years A.D. 1950 to 6050 B.C. These calibrations are based on measurements made to a precision of about 5 per mille (ie. about ±40 years), which was the best attainable during most of the past two decades.

Recently, radiocarbon dating laboratories have been established specialising in higher precision measurements than hitherto attempted. These may attain a precision of about 2 to 3 per mille (i.e. about ±20 years). An independent calibration of the radiocarbon time-scale is in progress at these laboratories using growth-rings from Douglass Fir and oak rather than Bristlecone Pine.

In Europe new dendrochronologies are under construction based on oak, and these are becoming comparable in length and quality to the Bristlecone Pine chronology. The growth-rings of oak and fir are much wider than those of Bristlecone Pine, and so larger quantities of sample are available, thus facilitating the higher precision measurements. The laboratories working together in this calibration programme are in Belfast, Groningen and Seattle.

Results from these laboratories show excellent agreement over extended periods, and there appears to be little need for the extensive use of statistical manipulations required for the more scattered and less precise Bristlecone Pine measurements. Individual sections of this work have been published and one of these has been used in the present work (Stuiver, 1982; Pearson, et al. 1983; Pearson and Baillie 1983).

It will be recalled that in his checks on the radiocarbon method Libby used the half-life of 5720 ±47 years. This, however, was soon altered, as a result of averaging several published determinations, to 5568 ±30 years, and this latter has become the accepted value and used as the basis on which all published "conventional" radiocarbon dates are calculated. The latest half-life determinations (Mann, et al. 1961; Watt, et al. 1961; and Olsson, et al. 1962) have yielded the half-life of 5730 ±40 years, in good agreement with Libby's original figure, and this is believed to be the most accurate value. Nevertheless, by agreement at the 5th International Radiocarbon Conference (Godwin 1962), and endorsed at later conferences, it was decided that 5568 years for the half-life would continue to be used in reporting radiocarbon dates in order to avoid confusion in the literature. It should be emphasised that this value is used exclusively in the newer calibrations which convert "conventional" radiocarbon dates to calendar dates, or to "dendro" dates.

13.7 Dates from el-Amarna

In many of the previous series of measurements on ancient Egyptian specimens the criticism has been made that the provenance of the samples has been insufficiently secure. Although the basic Egyptian chronology is believed to be well-founded, the credentials for any individual specimen to be dated may be less so. These are dependent upon the individual worker and upon the close observation of the material, its surroundings, and their accurate correlation with some identifiable event. Here is one of the frailties of the projects, for many workers have little or no training in chemistry, physics or botany. Yet some degree of proficiency in these is required unless the site investigated is otherwise foolproof. For reliable radiocarbon dating it is important to recognize when contamination with radioactive carbon from other sources is possible or probable - not only long rootlets from vegetation but also the percolation of carbon-rich ground water, or bicarbonates from the solution of limestone, even when from sources some distance from the site. In a land devoid of large timber it is necessary to be alert to the possible re-use of building materials. It is possible also that the wood of a tomb might be impregnated with carbonaceous liquids of diverse origin and age. With the larger timbers the inner pith could be centuries older than the outer growth-rings, but only the latter could be useful for dating the construction. The possibilities for choosing an appropriate specimen are many. Once the choice has been made the assignment of an historical date must be within close tolerances if the exercise is to have meaning. In the past much expense and effort have been wasted on dating inadequate samples.

The specimens reported on here, from a site within el-Amarna (specifically the Workmen's Village), appear to be exemplary as far as these problems are concerned. The city was constructed specifically for the Pharaoh Akhenaten (Amenophis IV) in the late Eighteenth Dynasty, and seems to have been occupied for only about fifteen years, with local extensions into the reign of Tutankhamun taking the limit possibly to about twenty years (cf. Chapters 1 and 9). The reign of Akhenaten is calculated to have lasted between about 1350 and 1334 B.C., with the earlier but less likely possibility of 1363 to 1347 (Wente and Van Siclen 1976; cf. Kitchen 1977-78). From the radiocarbon point of view this represents but a point in time in the long history of ancient Egypt. The site from which the samples were taken is small and isolated, and represents the remains of a midden probably deposited by the quarry workers early in the site's history, thus during the reign of Akhenaten rather than that of Tutankhamun. The samples originated from stratum number M10[83], a layer of earthy rubbish lying on the quarry floor (see Figures 6.3 and 6.4, pp. 85, 87). [1] The samples were lying on a stone base and were mixed with a brownish soil which was overlain first by a darker midden deposit, and then by a thick layer of sand. Rainfall in the region is extremely low, and the arid conditions have helped to preserve the organic materials largely unchanged. The conditions imply that significant contamination of the specimens is unlikely. From forty-eight separate samples collected, seventeen were too small for the present dating system assuming reasonable precision is required, and so were put aside until smaller counters become available. Other samples similar in nature were amalgamated to form three sufficiently large samples. Twenty-five were suitable for processing. These can be divided into two categories: (A): those that grew during a single season, and (B): those that integrated carbon over a number of years of growth. The classes were: Type A: leaf, fruit seeds, linen, coprolite, rush and rope; Type B: wood, charcoal, animal bone, horn and animal skin.

Type-A materials are liable to variable fluctuations of the radiocarbon content because of atmospheric variability, and so have "built-in" uncertainties that might amount to a century. There are, nevertheless, methods available for dealing with this type of sample. Type-B materials tend to integrate their carbon during their lifetime so that small pieces of wood or charcoal of the order of a decade in age are suitable. Similarly, bones of small animals are excellent specimens since the collagen also integrates the carbon intake over their lifetime. Horn and skin may be slightly more suspect and much depends on their carbon residence time.

It is essential that mass spectrometric measurements of the delta C-13 value are made for the samples so that a correction for isotopic fractionation may be performed. This is most important if accurate radiocarbon dates are required. With the limited resources available for this series it has been possible to date only five samples from the Type-B list. However, it is hoped that further work will be possible at a later stage.

13.8 Specimen preparation and processing

Despite the apparent lack of contamination, the specimens were cleaned thoroughly before the radiocarbon content was measured. Each was shaken in the ultrasonic bath to dislodge loose contaminants. The wood sample was

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[1] The samples were collected specifically for radiocarbon age determination on March 14th and 15th, 1982, during the excavation of the Main Quarry fill. The collection was made by the site supervisor, Martha Bell, in discussion with B. Kemp. Great care was taken to prevent contamination from wind-blown material and from debris falling from exposed strata, and subsequent handling was kept to a minimum.

powdered in a steel hammer-mill so that the chemical reagents would be more effective. It was leached successively three times with boiling dilute hydrochloric acid solution, boiling water, warm 0.5% sodium hydroxide solution and boiling water. Finally, it was washed with cold 50% hydrochloric acid and then water until the filtrate became neutral in reaction. The powder was dried in an oven overnight. The charcoal and animal skin were treated similarly. The horn sample, after powdering, was treated with cold 1% acid and alkali before washing thoroughly. The treatment of the bone was slightly more complex in that the protein fraction (collagen) was extracted for dating, and the main inorganic bone salt was destroyed. The bone was coarsely powdered and rinsed with cold 1% alkali solution and washed. It was treated with hot 8% hydrochloric acid for 20 minutes and the mixture poured into a large volume of boiling distilled water. This was kept hot and at pH 3 overnight. The residue was filtered through glass paper and the liquid evaporated to dryness. The collagen was converted by this process to gelatine which was used for dating.

The dried samples were oxidised in a bomb combustion unit (Switsur 1972) and the gases scrubbed in order to isolate very pure carbon dioxide. This was used to synthesize some pure benzene which contained all of the radioactive carbon from the original sample. A small portion of the carbon dioxide was taken for the mass spectrometric measurement.

13.9 Measurements and results

The synthesized benzene was made into a counting "cocktail" with the addition of butyl PBD and placed in a low level activity vial. The sample vials were placed in a liquid scintillation spectrometer and counted in sequence for one hundred minutes each. The activity measurements were repeated about thirty times until about 150,000 radiocarbon disintegrations were recorded for each sample. Various laboratory corrections were made to each of the measurements. The data were combined with those from measurements on modern standard samples and equipment background blank runs. Corrections were made for the isotopic fractionation effects. The results for the five samples studied are given in Table 13.1. In this the columns are as follows: column 1: a reference letter for this paper; column 2: the dating laboratory catalogue reference; column 3: the sample type and site number; column 4: the "conventional" radiocarbon age B.P. as defined above; column 5: the uncertainty of the age in years, this representing one standard deviation of the statistics, and including laboratory corrections and isotopic fractionation.

It will be observed that the conventional radiocarbon dates form a very close group with a difference of less than 65 years between the oldest and youngest. However, the maximum historical dates for the Amarna Period (including the reign of Tutankhamun) lie between about 1360 and 1325 B.C. (cf. Kitchen 1977-78), which are thus 3310 to 3275 years before A.D. 1950, the zero of the radiocarbon scale. There is a difference of some 260 years between the radiocarbon dates and the historical dates. This is typical of the situation found in dating Egyptian samples by this method. The calibration curves and

TABLE 13.1.

	LABORATORY REFERENCE	SAMPLE TYPE		CONVENTIONAL AGE B.P.	UNCERTAINTY YEARS
A.	Q-2401	WOOD	20	3030	35
B.	Q-2402	CHARCOAL	39	3055	35
C.	Q-2403	SKIN	12	3050	35
D.	Q-2404	HORN	35	3025	35
E.	Q-2405	BONE	37	3088	35

charts are intended to reconcile this difference. The results of the application of two different calibrations are demonstrated in the tables below.

Table 13.2 was obtained by using the charts given by Clark (1975). The comparison is made at one standard deviation. The rules given in the paper are not followed exactly since they were meant to apply to measurements having somewhat lower precision. This will affect the uncertainties but not the central value of the date.

TABLE 13.2. BRISTLECONE PINE CALIBRATION (CLARK)

SAMPLE	CALIBRATED AGE B.C.	UNCERTAINTIES +	UNCERTAINTIES -
A	1360	43	46
B	1390	39	45
C	1390	39	45
D	1352	44	46
E	1385	38	46

The figures should be rounded up to the nearest 10 years since without this the precision implied is spurious. The calibrated dates are certainly in the correct part of the time scale for el-Amarna. The central dates tend to be on the early side of the period, but taking the uncertainties into consideration the calibrated dates overlap the historical dates quite convincingly.

Table 13.3 was obtained using the new calibration of Pearson, et al. (1983), based on measurements of Irish oaks. This calibration is in the form of a graph (Figure 13.1) and the figures must be read from a fairly small scale. The numbers are rounded to the nearest 10 years.

TABLE 13.3. IRISH OAK CALIBRATION (PEARSON, ET AL.)

SAMPLE	CALIBRATED AGE B.C.	RANGE YEARS
A	1360 or 1285	1390 to 1260
B	1365 or 1305	1400 to 1260
C	1365 or 1305	1400 to 1285
D	1360 or 1285	1385 to 1260
E	1400 or 1330	1410 to 1300

It is important, in this instance, to explain the two calibrated ages and the need for a range of dates to express the uncertainty, rather than the usual $\pm$ term. It concerns the shape of the calibration curve in the region of

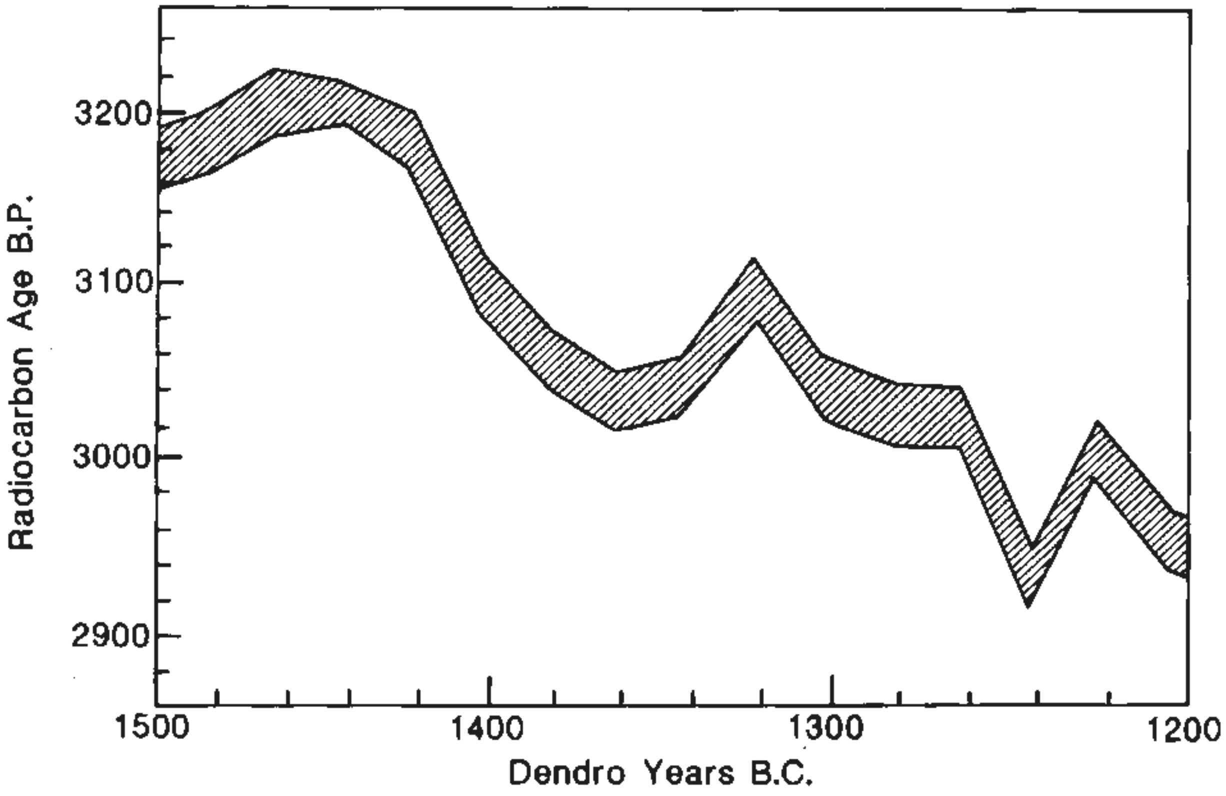

Figure 13.1. Calibration curve for radiocarbon dates based on measurements of Irish oaks (after Pearson, et al. 1983).

interest. A diagram of this portion of the calibration is given in Figure 13.1, taken from Pearson, et al. (1983), spanning the range 1500 to 1200 B.C. The curve was constructed from radiocarbon measurements of decade and bidecade samples of dendrochronologically dated oak. The hatched band represents the uncertainty of the calibration curve and this is estimated to be ±17 years at one standard deviation. It will be observed that during the Amarna Period there is a fluctuation of the atmospheric radiocarbon content which appears on the graph as an inverted "V"-shape extending from about 1340 to 1310 B.C. and flattening out on either side for a decade or so. This portion probably represents only three or four calibration measurements, and caution should be exercised in interpreting the resolution possible. To calibrate the radiocarbon dates, it is necessary to draw lines from the ordinate parallel to the dendro-year axis, and then to project them downwards on to the dendro-year axis. All the conventional radiocarbon dates in this series intercept this inverted "V", or pass nearby. Since the calibration curve is cut twice, there are consequently two possible dendro dates for each sample.

The calibrated uncertainties in the dates are obtained in a similar manner: the uncertainty quoted with the radiocarbon date is both added to and subtracted from the central date and the two results calibrated as above. The uncertainty used here must be the full value including the laboratory uncertainties and the uncertainty of the calibration curve itself (here ±17 years), and these must be combined in quadrature in the normal manner for independent variables. In the present work the curve is cut two or more times and it is necessary to take the extreme values which include the possible dates. Without further independent information, it is not possible to decide which of the pair of dates is the more likely, though in this exercise it might be assumed that the dates would be: A:1360, B:1365, C:1365, D:1360, E:1330 unless the quarry had been occupied subsequent to the desertion of the city. If a large piece of timber were available from the site so that several samples could be obtained for dating at known intervals apart, it should be possible to match the wiggles on the curve and so obtain secure dates.

It may be concluded from this series of dates, limited though it is, that the new calibration curve based on Irish oak is effective, at least in the portion of the time-scale studied, in that the calibrated conventional radiocarbon dates are consistent with the established historical chronology for the Amarna Period.

13.10 Acknowledgements

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