Outlier Archaeology: Bellona. A Preliminary Report on Field Work and Radiocarbon Dates

PART II.—RADIOCARBON DATING OF SIKUMANGO MIDDEN: SAMPLE ANU-608

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By HENRY A. POLACH*

INTRODUCTION

THE most reliable radiocarbon age determinations are obtained when ample organic material of a physically defined and recognizable form (i.e. wood, charcoal, shell, bone, etc.) is found in a well stratified site, where the time correlation of sample material and archaeological horizon in which it is found can be readily ascertained.

Neither of these criteria applied to the sample collected by Dr. J. I. Poulsen. The sample, the only organic matter available from the Sikumango midden which produced the potsherd, consisted of the entire contents of a dark band of midden earth. The fine-grained sandy soil was stained a dark charcoal grey with sooty, powdery charcoal. A large amount of fine shell fragments and calcareous debris and some fish bone fragments could also be identified. Small hair rootlets penetrated throughout the sample.

Without doubt the " sample " was contaminated by earth deposited above the midden. Hence the success of deriving an age for the occupation of the midden from such a material hinged not only on the evaluation of the degree of contamination, but also on the isolation of a suitable and relatively uncontaminated organic fraction which might provide a reasonable terminus post quem radiocarbon age.

Such an experiment in dating could not have been undertaken without detailed information about the site and the environment which fortunately was available from Dr. Poulsen's field notes. All reference to the nature, texture, physical and chemical properties and composition of soils on Bellona was obtained from a comprehensive and independent study made by Dr. Dalsgaard (1970).

SITE AND SOIL DATA OF RELEVANCE TO RADIOCARBON DATING

The horizon which on top of the Sikumango midden is some 20–30 cm. thick (Figure 4) is a dark to very dark brown sandy loam, friable, with a weakly developed granular structure and of low clay content. Incorporated is a relatively large amount of gravel, and very coarse to coarse sand, with light coloured oolitic phosphate granules. The organic carbon content of the top horizon covering the midden was 2·8% C, a figure that compares well with the organic carbon content of the topsoil in the

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vicinity of the village (2–3% C). The topsoil in the vicinity of the midden was neutral to slightly alkaline (pH 6·9–7·9) with relatively high (3–5%) $CaCO_3$ content.

Of particular relevance, when considering mobility, origin and distribution of organic matter within the soils, and hence also the midden, are two observations made by Dalsgaard.

Firstly, no carbonate differentiation could be observed or established within any of the eleven profiles studied by Dr. Dalsgaard at Bellona, and he concludes that carbonates must have leached out at the same rate from everywhere within these shallow, highly porous and permeable soils.

Secondly, Dr. Dalsgaard established that Bellona soils generally do not contain silica-bearing minerals that normally constitute the main part of a soil, and that soil profiles I and II (described in Dalsgaard's study) of particular relevance to and in the vicinity of Poulsen's site, contain little or no clay.

Dr. Poulsen's colour slides and field notes further show that over the midden only the top horizon was in an irregular but relatively smooth contact with the midden. The midden itself was in direct but highly irregular contact with the subsoil. The transition of the subsoil into the slightly surface-weathered limestone bedrock was often strewn with boulders protruding to the ground surface. This observation was also made by Dr. Dalsgaard, and applies generally to all sites at Bellona.

Radiocarbon Dating of Sample ANU–608

Sample Treatment

The sample, as submitted, was examined under binocular microscope using 10–30× magnification. With side illumination hair rootlets and other intrusive organic debris were clearly visible and were removed. This took several days and was followed by a further separation by flotation and wet sieving, resulting in a complete removal of all *visible* foreign material (rootlets, plant fibres, seeds, chitinous insect debris). The shell and bone debris were not present in sufficient amounts to constitute a dateable fraction. Therefore, no attempt was made to separate them and the limestone fragments physically from the sample as all these carbonate-bearing materials would be completely hydrolysed by the strong acid treatments that were to follow.

After hand picking, the sample (two separate 500 g. lots) was placed in a long-necked round-bottomed flask and cold 1N HCl was added in drops; evolving CO_2 induced foaming, being allowed to subside before new additions of acid were made. The CO_2 was swept out of the flask by a stream of purified oxygen and the CO_2 was recovered for dating as ANU–608a.

On completion of the acid evolution the soil suspension was filtered and washed and the soil was transferred into a 5-litre beaker. About 4 litres of 6N HCl were then added and the hydrolysis of recently comminuted organic matter, which would be partially decayed and hence not readily visible, was allowed to proceed overnight

just below boiling point. This step was repeated once more. Due to this severe acid digestion the carbohydrates and related substances (saccharides such as cellulose, pentosans and pectins) were broken down and solubilized. Demineralization of the soily sample also took place and acid washing continued until tests for Ca^{2+} and Fe^{3+}

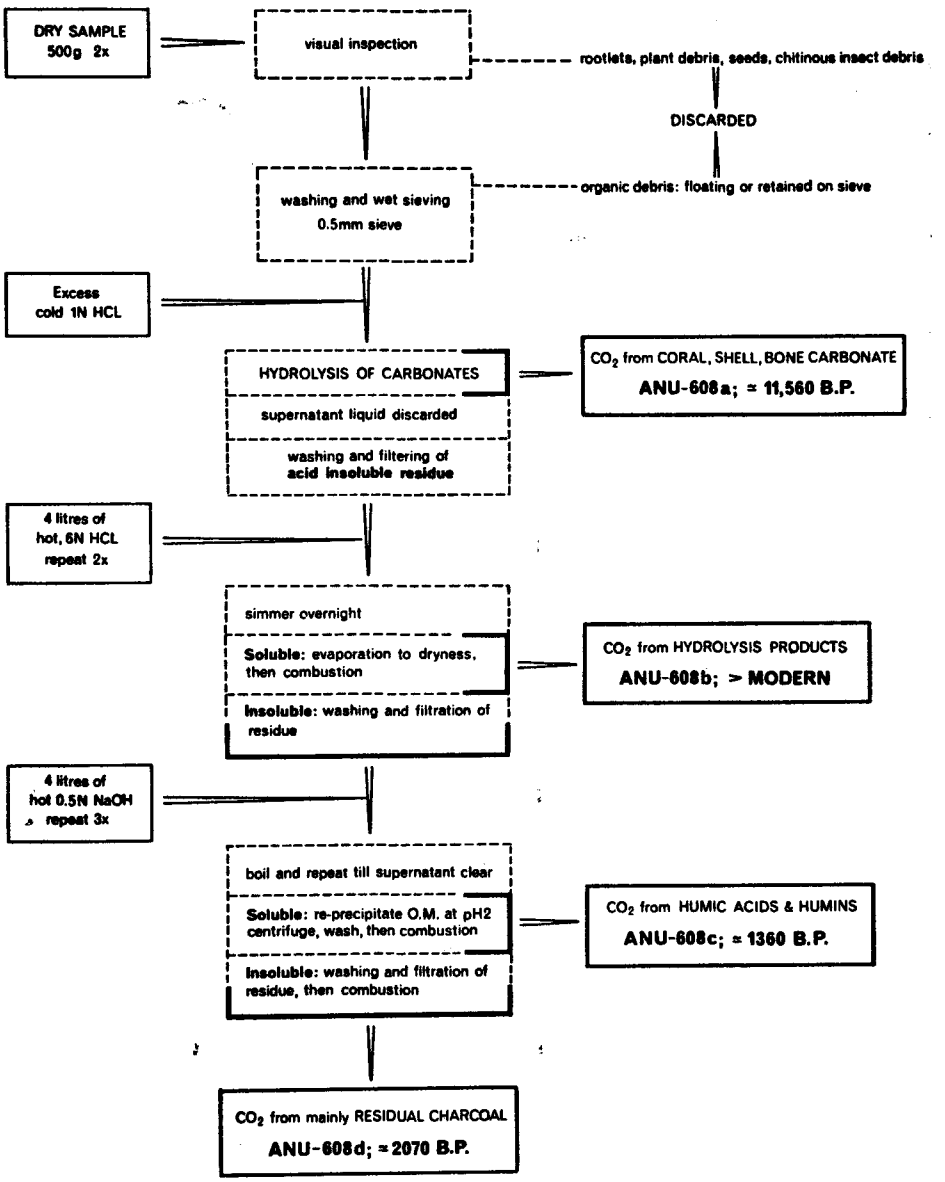

Figure 6.—Flow chart of treatment given and fractions isolated for radiocarbon dating from "sooty" soil sample, ANU–608, Sikumango mound, Bellona Island.

were negative. The hydrolysis products (i.e. soluble compounds) present in the brown but clear supernatant liquid, after evaporation under partial vacuum in a rotary-evaporator, yielded upon combustion ANU–608b.

The humus substances and products of advanced decomposition of organic residues (lignin, waxes and fatty acids, protein-like substances, humic acids, fulvic acids, humins) would not be rendered soluble, and were extracted by successive and exhaustive treatment with hot 0·5N NaOH. Humic acids and humins are soluble in hot alkaline solution and flocculate when their solution is acidified to a pH of 2–3 with HCl or H_2SO_4. The flocculants can be recovered by centrifugation, leaving a dark coloured but clear supernatant liquid containing mainly fulvic acids (Kononova, 1966). The isolated humic acids and humins were then dated as ANU–608c.

The acid-soluble fulvic acids and saponified fatty acids were discarded, as their age (anticipated to be *modern*) would not be of interest.

The NaOH-insoluble organic matter, presumed to be mainly charcoal from occupational activities (for example fireplace of Figure 4), yielded upon acid washing and combustion ANU–608d. An acid wash of NaOH-insoluble organic matter is necessary before drying and combustion, as otherwise the alkaline residues absorb CO_2 from the air. Air CO_2 activity is *ca.* 60% above the 1950 radiocarbon reference standard due to C^{14} produced by the testing of nuclear weapons. Any trace of residual CO_2, which would therefore contaminate the sample, is removed by an acid wash.

Figure 6 illustrates diagrammatically the adopted procedures.

Radiocarbon Dating of Chemical Fractions

The separation of the sample into fractions was carried out by H. A. Polach at the Institut für Bokenkunde (Soil Sciences), University of Bonn, Germany. The sample fraction CO_2 was recovered in two 1 m. long columns charged with 8% NH_4OH (ammoniacal) solution. The soluble ammonium carbonate, $(NH_4)_2CO_3$, was recovered as strontium carbonate, $SrCO_3$, after addition of strontium chloride, $SrCl_2$, to the boiling ammoniacal solution. The recovery and washing of the $SrCO_3$ was carried out on a glass sinter with CO_2-free, freshly boiled, distilled water, and the ammonia-free $SrCO_3$ was then dried in an oven prior to sending it in stoppered and sealed glass jars to the A.N.U. Radiocarbon Dating Laboratory in Canberra for dating.

In Canberra the $SrCO_3$ was acidified and the evolved CO_2 recovered and converted to benzene (Polach and Stipp, 1967; Polach *et al.*, 1972) and dated in a liquid scintillation spectrometer (Polach, 1969).

Mass spectrometric measurements of the stable isotopic composition of organic matter fractions from the soily sample were not carried out, as previous studies have established that there is little or no isotopic fractionation during saprophytic organic matter degradation processes by microorganisms, or chemical fraction extraction methods (Polach *et al.*, 1969; Rafter and Stout, 1970).

The results of radiocarbon age determinations are listed in Table 1.

TABLE 1

Radiocarbon Ages of Fractions Isolated from Sooty Soil Sample, Sikumango Mound, Bellona Island

Lab. Code and No.	Fraction Dated	Age ± Error B.P.	Comment
ANU–608a	Carbonate	11,560 ± 170	Shell, coral and bone carbonate
ANU–608b	Hot 6N HCl hydrolysis products	+32·3 ± 1·7% >MODERN*	CO_2 from recently comminuted, partially decayed OM†
ANU–608c	Hot 0·5N NaOH exhaustive extract	1360 ± 120	OM in advanced stage of decay and soil humus substances
ANU–608d	Acid and alkali insol. residue	2070 ± 80	Mainly residual charcoal from occupational activity

* Post-1950 activity and "age".
† Organic matter (plant, animal and insect).

Interpretation of Radiocarbon Results

1. ANU–608a. Total carbonate age: 11,560 ± 170 B.P. is of no significance to the midden dating problem as the age is derived from an unknown mixture of weathered limestone and man-introduced carbonaceous material. The limestone bedrock is older than Pleistocene (Grover, 1960), but weathering processes would introduce atmospheric CO_2 (dissolved in rain or as bicarbonate in solution) which would make its age appear within the C^{14} range. Indeed, only 1% addition of modern carbon to a sample which contains no radiocarbon at all will result in an apparent age of 37,000 B.P. (cf., for example, Polach and Golson, 1966; and others). Further addition of relatively recent man-introduced midden debris, shell and fish bone fragments, not found in the surrounding soil, possibly make the resulting C^{14} age even younger. As we could not isolate from or relate proportions of shell and bone fragments to the soil carbonates, the result must be ignored as it is not relevant to the age of the midden.

2. ANU–608b. Acid hydrolysis products age: >MODERN (ΔC^{14} = +32·3 ± 1·7%), gives the "age" of the contaminant, i.e. the recently comminuted, but now partially degraded organic matter, which thus escaped visual inspection and separation. The >MODERN, i.e. *contemporary*, activity can be related directly to the increase of C^{14} in the atmosphere due to nuclear weapons testing.

Solubilization of cellulose is a standard technique developed by wood chemists (Wise and Jahn, 1952). However, Olson and Broecker (1958) were the first to apply the techniques in a radiocarbon dating laboratory in order to purify or fractionate samples submitted for dating; they suggest a selective hydrolysis of cellulose in cold 41% HCl, a procedure closely related to the technique described in our previous section on sample treatment. Our own experience as related to the extraction and isolation of dateable wood fractions (Polach *et al.*, 1969; McDougall *et al.*, 1969)

confirm the value of the procedure. Jenkinson (1968), in a study of the rate of decomposition within a soil containing radioactively labelled plant material, further found the acid hydrolysis most effective in extracting from soils the labelled organic carbon which through partial decay had recently (within the preceding four years) been comminuted to a soil (Jenkinson, 1968: 37). We therefore can postulate that *intrusive* and *mobile* organic matter constituents (Polach and Costin, 1971) would be hydrolysed (hence removed) from the sample we wish to date.

The " age " alone of the hydrolysable fraction, i.e. *contemporary*, is proof that we have achieved our aim. It remains only to establish how well we have achieved this. No direct measurement is possible for the site studied, as only one sample was available at Bellona. However, Rafter and Stout (1970), in a study of forest and grasslands C^{14} uptake since nuclear weapons testing, have shown that the accumulation of *contemporary* carbon within a soil profile is relatively small if compared to the bulk carbon content of the soil. We therefore conclude that the residual amount of recent organic matter in our remaining fraction would be negligible.

3. ANU–608c. Exhaustive caustic extraction product age: 1360 ± 120 B.P. is the " apparent age " of material presumed to be foreign to the deposits we wish to date. The age is the result of the compound C^{14} activities of humic acids and humins which are solubilized by hot NaOH treatment (Scharpenseel *et al.*, 1968) and lignins which have the same chemical affinity as soil humus substances (Kononova, 1966), and would therefore also be mostly solubilized. The observed " apparent age " thus represents the *mean residence time* of non-hydrolysable soil organic matter, as first postulated by Paul *et al.* (1964) and quantified by Geyh *et al.* (1971).

It is important to note that after demineralization of our sample by the preceding hydrolysis with large quantities of strong acid, the generally insoluble calcium humates and fulvates would also be solubilized as their sodium salts, due to action of succeeding exhaustive (repeated) NaOH treatment. In the absence of clay minerals and siliceous minerals, the otherwise insoluble mineralized humates would also be rendered soluble (Tyurin and Gutkine, 1940), thereby possibly reducing to insignificant amounts the fraction of soil organic matter that would be carried over into the residual fraction.

4. ANU–608d. The residual fraction age: 2070 ± 80 B.P. represents mainly the black " sooty " charcoal within the sample which Dr. Poulsen came across exclusively during his excavation of the midden material. Dr. Dalsgaard also confirms that no such material was evident in any of his eleven profiles studied on Bellona Island. As fish bone and shell fragments were also associated only in the material submitted for dating, it is fair to assume that the residual fraction is mainly charcoal dating the activities of man at this site. This is further confirmed by the increase in organic carbon content of similar midden material which Dr. Poulsen had investigated. The analysis gave 6·9% C, 7·7 %and 7·1% C, whilst the top horizon of the Sikumango mound gave only 2·8% C.

The lack of $CaCO_3$ differentiation throughout the island as observed by Dr. Dalsgaard, and the paucity of organic carbon in the subsoil, illustrate the lack of

mobility of soil organic matter in the presence of free and excess calcium in this highly porous and permeable soil. This factor played a significant role in the evaluation of validity of radiocarbon ages from this site, and it appears plausible to state with a considerable degree of confidence that the age of human occupation of the Sikumango site lies between the limits of 1910 and 2230 B.P., i.e. within two standard deviations of the quoted result, ANU–608d (2070±80 B.P.).

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