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RADIOCARBON DATING, SOME GUIDANCE FOR USERS

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Three of the most common questions archaeologists wishing to submit C14 samples ask are:

What size must the sample be?

How many samples may I submit?

Is such and such a material all right for dating?

This note is an attempt to give some guidance on these three questions. The answers are directed towards the requirements and experience of dating New Zealand samples at the D.S.I.R. laboratory at Gracefield in Wellington. The recommendations may not therefore be universally valid, particularly where other laboratories are involved.

Sample size

To count the disintegration of carbon atoms of the carbon 14 isotope samples have to be turned into a gas. At the D.S.I.R. carbon dioxide (CO₂) is the gas commonly used. The method of production of this gas varies with the sample material. The common archaeological materials are listed below.

Charcoal, wood, fern stalks, leaves and bark. These samples will burn. The CO₂ is produced by burning the sample in a stream of oxygen.

Shell. The CO₂ is generated by adding phosphoric acid to the sample. Shells are made principally of crystalline calcium carbonate (CaCO₃). The effect of acid is to release carbon dioxide while dissolving the rest of the shell material.

Bone, as with shell contains calcium and other carbonates which can release CO₂ on dissolution with acid. This material has proved to be unreliable for dating because of the tendency of this material to be contaminated by more modern carbon. However, acid dissolution leaves behind an acid insoluble fraction of the bone. This is usually labelled collagen. Collagen is a complex protein found in bones which is relatively resistant to decomposition. When this material is burnt to

Material	Weight grams	
Pure carbon	11	
Charcoal	22+	(a)
Wood	27	
Shell	85	
Bone	1,000	(b)

(a) This size charcoal sample is usually sufficient but in cases where more is available it is prudent to send up to 120 g.

(b) Samples smaller than this can be satisfactory. Sample yield can be checked on gram sized samples and the minimum sample size determined. In many cases smaller sizes would be desirable to conserve valuable specimens.

TABLE 1. Preferred minimum weights for C14 samples.

to produce CO₂, it yields much more satisfactory dates. However, in general, the older bone is, the less collagen it will contain.

The three classes of materials above yield different amounts of carbon dioxide for a given weight of sample.

The more carbon that the laboratory can count the lower the counting error will be and the lower the reported error range for the age. The laboratory has a variety of counting vessels which it uses for different sized samples holding differing amounts of gas. For samples of the age range of New Zealand prehistory the largest, if filled, can get the accuracy of a date down to around ± 42 years. Figure 1 shows graphically the best errors that can be achieved if the counter is filled.

Filling the counter needs the sample sizes shown in Table 1. Not having a large enough sample need not be fatal but will result in a date of lower precision. The precision which might be achieved by a smaller sample can be estimated using the following formula, for m_{ideal}/m greater than 1 only:

$$e = \pm E (m_{ideal}/m)^{1/2} \text{ years} \quad (1)$$

- where e is the estimate of the error which can be achieved, E is obtained from Figure 1, m_{ideal} is the ideal sample weight in grams given in Table 1, and m is the actual sample weight in grams.

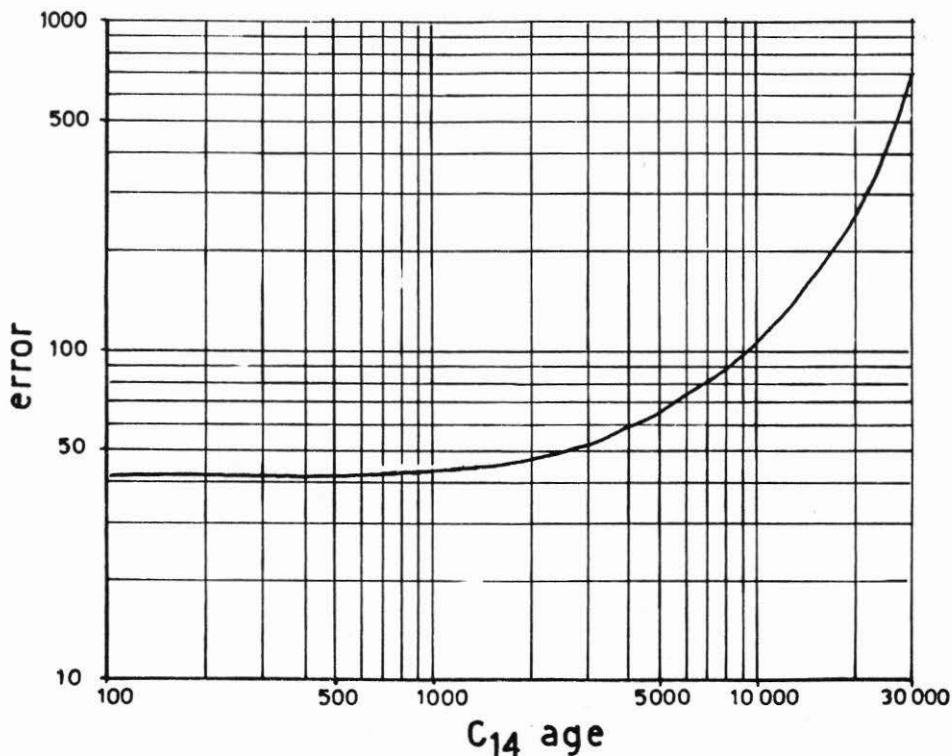


FIGURE 1. Graph shows the best error which can be achieved for samples of different age B.P. Both scales are in years with a log conversion. Based on filling the largest counting vessel and a 100 minute count.

Example. A shell sample believed to be of the order of 2,000 years old weighs 30 g. What error can be expected? From Figure 1 for a 2,000 year old sample, $E = 47$, from Table 1 $m_{ideal} = 85$ g, therefore by equation 1

$$\begin{aligned} e &= \pm 47(85/30)^{\frac{1}{2}} \\ &= \pm 79 \end{aligned}$$

There may well be cases where in attempting to falsify propositions that error ranges of greater than ± 100 are acceptable. Generally though, in a New Zealand context they are unlikely to be acceptable.

How many samples

The D.S.I.R. laboratory provides an enviable dating service to New Zealand archaeologists. It is actively involved in checking modern standards using New Zealand and the Pacific materials of known age; it routinely corrects dates for fractionating; its counting system has checks against spurious counts from Radon disintegration; and it supports a consulting committee to advise it on archaeological problems. Few commercial labs offer any of these services.

The cost of dates from a commercial laboratory is of the order of \$200. The value of dates from the D.S.I.R. is much higher.

Now clearly it is possible to abuse a free service. The laboratory is making a substantial investment in determining the age of samples and it is only fair that the results should relate to an investigation which is in itself a substantial investment of effort by archaeologists. There can be exceptions of course, say where erosion exposes a section through a site which has clear cultural significance and which may only involve a small effort to record and report. Dating in this circumstance may be a large part of the investigation. In contrast, the use of C14 dating as a prospecting tool to find sites of a certain age, or in an investigation of the age of first settlement of an area, centres on age as an end in itself, rather than one aspect of a wider range of evidence. Such a use of the service can only rate a low priority.

Narrowing the question down to how many samples from one site focuses attention on the site itself. A site with clear stratigraphy and evidence of a long time span can repay a lot of dating. Dates spread through the occupation sequence can result in a lot of events in the site being closely dated. Dates from a stratigraphic sequence can support each other for not only can a layer be dated but dates for layers above and below can support the age by defining limits. However, often material in sites is not in sealed stratigraphic contexts. It is not clear whether the length of occupation is years

or centuries, or if evidence on one part of a site is contemporary with evidence from another part or earlier or later than that part. Is carbon dating a tool to attack these problems? In the sort of time differentiation we would wish, it probably isn't unless a lot of dating is done.

The nature of the process is such that it is not likely that any amount of dating can prove that the real time extent of occupation of a site is less than 200 years. This is well short of demonstrating contemporaneity. It can only support an argument for contemporaneity developed from other evidence. I would suggest that given a choice of dating more samples from one site to demonstrate contemporaneity or dating samples from new undated sites, the latter should get priority.

Still looking at sites, there are many strategies possible in selecting which parts of a site to excavate and how they are excavated. One could tackle a site with a lot of discretely separate sub-investigations and some sites or problems might demand this. However, beware as dating is no substitute for stratigraphic control and any proposed dating programme which has its justification in propping up a poor excavation strategy can expect little priority.

The question also arises of running more samples from a site when one or more from an earlier batch are away outside expectations. It is an unfortunate fact that new dates will not make the old ones go away and trying to drown unwelcome data in a sea of more acceptable results is a response more satisfying to the ego than the intellect. This sort of problem is better tackled in the first instance by a thorough review of the site and the samples to see if a rational explanation can be found. If this explanation can then be tested by more dating with some benefit either to knowledge of the site or knowledge of C14 dating then more dating can be justified.

What to use for samples

Archaeologists in the happy situation of having a choice can read on! In some circumstances, however, painstaking collection may yield an adequate sample of a more suitable material than an easily collected unsuitable one.

The following lists some advantages and disadvantages of different materials.

1. Marine shell. Generally a highly satisfactory material, being increasingly used and producing highly consistent results.

Contemporaneity. Use of food species should ensure this. Check though for evidence of attrition from dead shells wearing on beaches, or growth of encrusting marine organisms after death. Beware of redeposited midden on sites with intense earthworks, and shell from beaches 'borrowed' as a soil conditioner for horticulture.

Standards. Avoid mud snail (*Amphibola crenata*) which has proved anomalous. Estuarine species in limestone areas could cause problems.

Contamination. This can be investigated by checking recrystallisation by x-ray. Recrystallisation indicates contamination has had the opportunity to occur. This check is carried out by the lab.

Sample treatment. Wash (ultrasonic is good if available) and air dry. Do not use detergents to aid cleaning. De-ionised water is best if to hand, otherwise tap water is acceptable.

2. Grass, seeds, leaves of woody plants.

Contemporaneity. Obviously excellent.

Standards. These sorts of materials suffer suspicion of having been affected by the 11 year cycle of atmospheric C14 which has been identified for the early 20th century and could have existed before (see Law, 1971).

Sample treatment. Air dry. Do not apply preservative treatments to C14 samples.

3. Charcoal identified as from new wood or short lived species.

Contemporaneity. Good. Note that experience is showing that samples are almost always mixtures of three or more species. Beware of fossil charcoal from earlier human or natural fires.

Standards. There are no major problems in this area.

Contamination. Humic acids are likely contaminants but the removal of these is controversial. For samples from New Zealand human prehistory the likely order of disturbance does not justify the attempt at the present stage of knowledge.

Sample treatment. Clean sample, sieve out sand, pick out shell, bone, stones, remove as much soil as possible. Air dry. Never oven-dry samples as this can 'activate' the charcoal and make it prone to absorbing atmosphere-borne organic compounds.

At the identification stage the more suitable sample constituents can be sorted from large samples. Size sorting can cause differentiation of species but is not recommended 'blind' as the large-small differentiation need not equilibrate to age at death.

4. Unidentified charcoal.

Contemporaneity. This is your guess. Experience suggests samples are commonly of charcoal from 200 year-old wood, and can be up to 500 or 1,000 years older than the burning of the sample.

5. Marine fish bone.

Contemporaneity. This should be excellent.

Standards. Avoid pelagic fish which could draw their food from other than New Zealand waters.

Contamination. This is little known at present.

Sample treatment. As shell.

6. Terrestrial bone other than human.

Contemporaneity. Beware of industrial bone. Food species should be best.

Standards. Land animals with some marine input to their diet may cause problems (ducks, dogs). Good species are those with wholly insect, nectar, plant foliage or other terrestrial animals in their food chains. Moa is quite acceptable.

Contamination. There is reason to doubt that the acid-insoluble portion of bone (the collagen) is necessarily being uncontaminated but this is an unsolved problem.

Sample treatment. Clean and air dry.

7. Human bone. There are of course ethical problems to be considered in using such materials. With other doubts on standards and contamination, human bone cannot be regarded as a very satisfactory material.

Contemporaneity. Obviously primary.

Standards. There are major doubts in this area for people with access to marine foods.

Contamination and treatment. See above for terrestrial bone.

8. Marine mammals and birds. The comments above on other bone material generally apply in respect of contemporaneity.

Standards. Animals which draw their food in the tropical or Antarctic seas are suspect, so avoid widely travelled species.

9. Soil. In most circumstances soil remains a 'living' entity continuing to gain and lose carbon to and from the present world. Specialist advice should be obtained for soil samples.

10. Peat.

Contemporaneity. There should be few problems in this area but the rate of accumulation of the sample should be considered.

Standards. Again there are no problems for this sort of material.

11. Bark. Bark from large trees can be considerably 'older' than the wood immediately under the bark. This is the case through the bark having grown with the tree and some at least of the material now on the outside originating when the tree was much younger. Material from long-lived trees is better avoided.

12. Ash. Ash from fires can yield carbon for dating - even when no charcoal is evident. However it is not clear what in fact has been dated as the carbon could be from one of several origins. Unless it is all that is available this material is better avoided.

Acknowledgement

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References

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