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## <sup>14</sup>C VARIATIONS FROM TASMANIAN TREES— PRELIMINARY RESULTS\*

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**ABSTRACT.** Huon pine is endemic to Tasmania. It has well-defined annual rings, may live for over 2000 years, and is particularly resistant to decay. Celery-top pine has similar characteristics and may live for 800 years. As part of a multi-disciplinary study of these trees and their habitat, a simple wood pretreatment method for isotope analysis is described. The solvent-acid-alkali-acid sequence yields a value of  $\Delta^{14}\text{C} = -16 \pm 6\%$  for AD 1941-45 Huon pine heartwood;  $\Delta^{14}\text{C}$  for extracts containing various proportions of post-AD 1955 carbon are also presented.  $\Delta^{14}\text{C}$  measurements on super-canopy and sub-canopy leaves from Celery-top pines are compared and used to place an upper limit of 10% on the amount of sub-canopy  $\text{CO}_2$  assimilated by sapling leaves, originating from decaying litter-mass.  $^{14}\text{C}$  ages from well-preserved logs illustrate the potential for a continuous Holocene chronology from 7400 years BP to the present. A 12,000-year-old Celery-top log has also been found.

### INTRODUCTION

Huon pine (*Dacrydium* now *Lagarostrobos* (Quinn, 1982) *franklinii*) is a long-lived species confined mainly to river valleys in western and southern Tasmania. Individual specimens may live for more than 2000 years, while well-preserved logs of even greater antiquity may be found either on the surface or buried, since the wood is particularly resistant to decay. Celery-top pine (*Phyllocladus aspeniifolius*) has similar characteristics and may live for 800 years. The presence of well-defined annual rings in both Huon and Celery-top pines and coupling between variations in xylem growth and microclimate permit cross-dating and construction of long chronologies of ring-width series anchored in the present. Since their annual rings may be chemically treated so that only material assimilated in the year of growth remains, they can provide a long, accurate record of atmospheric variations in  $^{14}\text{C}$  for the southern hemisphere.

### SITE DETAILS

Wood collections were mainly carried out in a small area of rain forest on the middle reaches of the Stanley River. The area was lightly and selectively logged in the past so that cross-sections, 5 to 20cm thick, are easily obtained from tree stumps and fallen timber. The area also contains many ancient logs buried under 2 to 3m of river sediments. Some are exposed where the modern river cuts through the deposit; others were found by excavation. The site is within 23km of the Tasmanian West

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Coast in an area which, apart from some bushfires, was almost free of human influence. Winds are predominantly from the southwest, so that the composition of the troposphere largely represents that over the southern oceans. Rainfall is high, in excess of 2000mm per year. Huon pine is generally restricted to areas of permanent surface water (*ie*, along water courses or in swampy areas, eg, Pedley, Brown, and Jarman, 1980), whereas Celery-top pine is more widespread.

#### DENDROCHRONOLOGY

Ogden (1978) points to the relative complacency of Huon pine growth-ring series, thought to be induced by moist habitat. The absence of water stress in the Huon pine environment may be advantageous in that it minimizes carbon isotope fractionation arising from concentration gradients across the stomates (Francey and Farquhar, 1982). Preliminary studies on Huon pine from the Riveaux Creek area by T Bird have demonstrated infrequent occurrences of "missing" and "multiple" rings, which coupled with some ring-width variation, permit successful cross-dating and chronology development. La Marche and Pittock (1982) found Celery-top pine grew more after winters which were warmer and drier than average, but suggested that the variables tested (temperature and precipitation) were a measure of available light. Ring-width variation in Huon pine may also be due to changes in integrated light intensity in these wet forests, since Huon and Celery-top pine cross-date in many years.

#### PRETREATMENT METHODS

We developed a simple pretreatment method by which a suitable fraction of wood can be separated for isotope analysis. Although isolation of pure cellulose would have been a sure way to proceed, preparation time would have been excessively lengthy.

The remarkable resistance of Huon pine to decay (Millington *et al*, 1979) is due to the presence of oils and resins which inhibit the activities of micro-organisms. Since these oils and resins are mobile within the wood structure, it is important that pretreatment removes them. The de Vries method, of sequential acid, alkali, and acid extractions, when used on wood is somewhat successful in removing unwanted, mobile fractions. However, the success of the method with non-polar fractions such as oils and resins is limited because only highly polar water solutions are used. Following the suggestions of Head (*ms; pers commun*) we decided to supplement this method with a series of solvent extraction steps to remove these fractions. The acid and alkali solutions would ensure that those fractions such as hemicelluloses, which can be made water-soluble, were removed. The alkali extraction would also break down and remove the low molecular weight lignins. The residue would be composed principally of cellulose but include the high molecular weight lignins.

A Huon pine from the Gordon River area was selected to test this supplemented method. Wood was taken from the AD 1941-45 growth rings that, in this tree, had already undergone the sapwood-to-heartwood conversion. The cellulose of these rings would not have any post-AD 1955

“bomb-generated” <sup>14</sup>C in it but some heartwood extractives and the mobile fractions would show much higher <sup>14</sup>C concentrations.

Pretreatment was tested on 0.2mm thick wood shavings, by sequentially boiling and filtering, using: 1) 2:1 benzene:ethanol solution, 2) ethanol, 3) water, 4) 2M hydrochloric acid, 5) 2% sodium hydroxide (twice, using a fresh solution each time), 6) 0.1% phosphoric acid, and then 7) washing with distilled water until neutral. The last four steps parallel the de Vries method. Two subsamples of the finely shaved and homogenized wood from these rings were produced. One subsample was given the full treatment while the other was treated by the equivalent of the de Vries method alone.

The results of <sup>14</sup>C and <sup>13</sup>C determinations are shown in figure 1, where the extract labeled *Solvent* was produced by amalgamation and evaporation to dryness of the extracts from steps 1 and 2; *Acid* from steps 3 and 4, and *Alkali* from the acidified filtrate of step 5. Insufficient material was generated in either steps 6 or 7 to warrant separate carbon isotope measurements. *Residue* refers to material remaining after step 7. The relatively high  $\Delta^{14}\text{C}$  for the solvent extract ( $318 \pm 18\%$ ) is slightly less than that for contemporary air at the time of felling ca 1974 (ca 400%; Polach and Singh, 1980), implying that some of the extract was formed prior to 1962, but with a high proportion of more recent material, as expected from the known mobility of resins and oils in wood. If the solvent extraction steps are omitted, some, but not all, of this fraction finds its way into

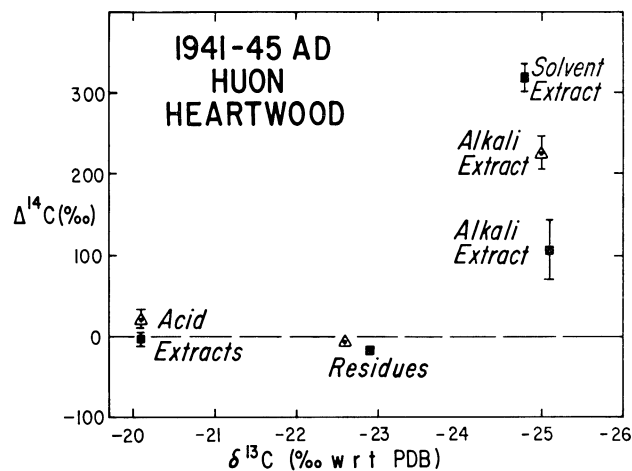


Fig 1. Carbon isotope measurements on chemical fractions of Huon pine wood from the AD 1941-45 growth rings.  $\delta^{13}\text{C}$  values are relative to the PDB standard;  $\Delta^{14}\text{C}$  values are normalized to  $\delta^{13}\text{C} = -25\%$  and corrected for radioactive decay (Stuiver and Polach, 1977). The wood sample was divided and subjected to different pretreatment steps; results from the full method which included solvent extraction are shown as ■, whereas results for the second portion which was treated only with acid-alkali-acid steps are shown as  $\Delta$ . Uncertainties are generally the size of the symbols ( $\pm 0.1\%$  for  $\delta^{13}\text{C}$ ,  $\pm 6\%$  for  $\Delta^{14}\text{C}$ ) except where indicated by error bars. Full pretreatment extracts more post-AD 1955 carbon and yields slightly less residue with lower <sup>14</sup>C activity and more negative  $\delta^{13}\text{C}$ .

the alkali extract; some of the oils and resins can be made water soluble by basic but not acidic hydrolysis. Alkali extraction also removes another component, containing mainly lignin. As the sapwood-to-heartwood transition involves deposition of contemporary carbon, the  $\Delta^{14}\text{C}$  of  $107 \pm 37\%$  (alkali extract, after solvent extraction) indicated that the transition on the AD 1941-45 rings occurred within a few years centered ca 1958 (see Polach and Singh, 1980). Alternative explanations involve small but significant contributions to pre-1956 material by carbon of more modern origin. The  $\delta^{13}\text{C}$  value indicates that the acid extract contains a component which is clearly different to those in the alkali and solvent extracts. Its  $\Delta^{14}\text{C}$  of  $-2 \pm 9\%$  (after solvent extraction) indicates that it antedates the alkali extract. It probably contains principally hemicelluloses.

The residue, (*Residue*, fig 2) after full pretreatment, gives a  $\Delta^{14}\text{C}$  value of  $-16 \pm 6\%$ , in good agreement with results from the northern hemisphere (Stuiver and Quay, 1981). This implies that the method yields a residue which is essentially free from components more than 10 years younger than the rings themselves. In contrast, the residue produced from the de Vries method alone gives a  $\Delta^{14}\text{C}$  value of  $-5 \pm 6\%$  indicating that post-AD 1955 components were not completely removed. This conclusion was previously reached for other species (Olsson, 1980).

#### $^{14}\text{C}$ OF CONTEMPORARY LEAF MATERIAL

If canopies retain a significant proportion of  $\text{CO}_2$  from decaying humus and litter, then significant levels of 'old' carbon might be re-assimilated and laid down in tree rings. To test this hypothesis, leaves were taken from a mature Celery-top pine (with all foliage above canopy height) and from a 2m sapling. The results of the  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  determinations are given in table 1.

Due to the steadily declining  $\Delta^{14}\text{C}$  in the atmosphere (at ca  $16\%$  per year since 1965) any contribution to sub-canopy  $\text{CO}_2$  from decaying vegetable matter would increase its  $^{14}\text{C}$  activity. For an anticipated carbon turnover time of 1 to 2 years for litter mass in this environment (based on data of Ajtay, Ketner, and Duvigneaud, 1979 and R M Gifford, pers commun), sub-canopy  $\text{CO}_2$  would be a mixture between  $\text{CO}_2$  of contemporary activity (taken as  $293\%$ ) and  $\text{CO}_2$  with  $\Delta^{14}\text{C}$  in the range 309 to  $325\%$ . In these circumstances, the  $1\sigma$  upper limit of the sub-canopy  $\Delta^{14}\text{C}$ , *ie*,  $295\%$ , represents an upper limit of ca  $10\%$  as the proportion of sub-canopy  $\text{CO}_2$  originating from litter and assimilated into the sapling leaves at 2m. Full details are given in Francey *et al*, (Isotopes in tree rings—Stanley River Collections 1981/82: CSIRO Tech paper, ms in preparation).

TABLE 1  
Carbon isotope measurements on Celery-top leaves  
above and below canopy, 1980/81 season

Tree	SUA No.	Relationship to canopy	$\delta^{13}\text{C}$ (‰) PDB	$\Delta^{14}\text{C}$ (‰)
SRT 74	5008	Below	-29.6	$286 \pm 9$
SRT 2	5009	Above	-24.0	$293 \pm 7$

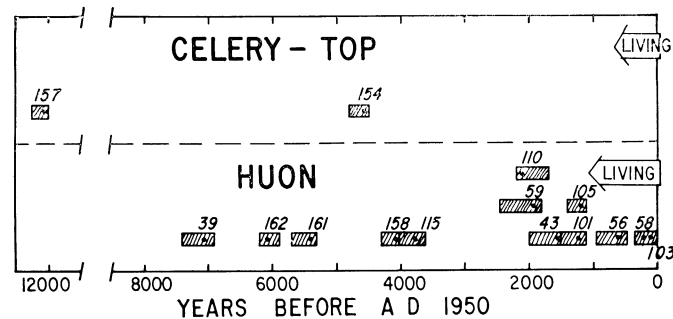


Fig 2. Approximate age ranges of preserved logs from which cross-sections have been collected at the Stanley River site. Numbers refer to our site catalogue, ● the calibrated <sup>14</sup>C ages (Klein *et al*, 1982) and ▨ the ages spanned by the rings in section. Many other buried logs have been sampled, but not yet dated.

#### POTENTIAL OF THE TASMANIAN PINE RECORD

The ages of some of the logs and stumps sampled in the field-work area are shown in figure 2. The outside of one Huon log (SRT-39) has a <sup>14</sup>C age of 6190 ± 60 yr bp (SUA-5004) corresponding to a calendar age of ca 7100 yr BP. It would appear likely in view of the well-distributed ages of the other samples, that wood with all ages between 7000 yr BP and the present will ultimately be found. The discovery of a Celery-top log with a <sup>14</sup>C age of 12,000 yr bp gives rise to the tantalizing prospect of an even longer record, especially since, though shorter-lived, Celery-top is easier to cross-date. A chronology based on Tasmanian pines stretching back to the late Pleistocene may be attainable.

#### ACKNOWLEDGMENTS

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