BETA ANALYTIC INC.

RADIOCARBON DATING SERVICES

Dr. JERRY J. STIPP Dr. MURRY A. TAMERS **CO-CHAIRMEN**

DARDEN G. HOOD, P.G. General Manager

RONALD E. HATFIELD Laboratory Manager

CHRISTOPHER PATRICK TERESA A. ZILKO-MILLER Associate Managers

March 23, 1995

Dr. Michael D. Hylland Utah Geological Survey Applied Geology Program 2363 South Foothill Drive Salt Lake City, Utah 84109

Dear Dr. Hylland:

Please find enclosed the results on material recently submitted for radiocarbon dating. This package includes the final date report, a cover form-letter outlining our analytical and reporting procedures, a glossary of pretreatment terms, calendar calibration information, billing documents (containing balance/credit information and the number of samples submitted within this years discount period), and peripheral items to use with future submittals. In future submittals, if there are special considerations with regard to the analyses, you will receive an additional personalized cover letter. In the absence of a personal letter, the analyses proceeded normally without special notice.

As we discussed, only one sediment was analyzed. A large portion of the sediment was analyzed radiometrically after applying acid washes (potential secondary organic acids not removed). A second, much smaller fraction, was analyzed by AMS after full pretreatments (secondary organic acids removed). The observed results are the opposite from that normally expected from secondary organic acid contamination. Removal of secondary organic acids should yield an older date. In this case, the fully pretreated AMS fraction gave an older date. This indicates the sediment contains multiple components with differing solubilities in the alkali, and wildly differing in C14 age.

As always, if you have any specific questions, please do not hesitate to fax, e-mail, or call us.

Sincerely, Darden Hood

4985 S.W. 74 COURT, MIAMI, FL, 33155 U.S.A. TELEPHONE: 305-667-5167 / FAX: 305-663-0964 / E-MAIL: beta@analytic.win.net

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Please note in your records that results are reported within "business days". A schedule card has been included in this package. It also contains a convenient list of quantities recommended for dating.

As always, if you have any specific questions, please do not hesitate to fax, e-mail, or call us.

Sincerely,

BETA ANALYTIC INC.



DR. J.J. STIPP and DR. M.A. TAMERS

REPORT OF RADIOCARBON DATING ANALYSES

Sample Data Measured C14 Age Beta-80451 SAMPLE #: FST2-RC2 ANALYSIS: radiometric-standard	C13/C12 Ratio	Conventional C14 Age (*)
C14 Age Beta-80451 3650 +/- 70 BP -: SAMPLE #: FST2-RC2 ANALYSIS: radiometric-standard	Ratio	C14 Age (*)
SAMPLE #: FST2-RC2 ANALYSIS: radiometric-standard	25.0* 0/00	0050 · / 70+ 00
ANALYSIS: radiometric-standard		3020 +/- /0* Bb
MATERIAL/PRETREATMENT:(organic sediment): acid	washes	_
Beta-80493 1770 +/- 50 BP -2 CAMS-18678	26.2 0/00	- 1750 +/- 50 BP
SAMPLE #: FST2-RC2 ANALYSIS: AMS MATERIAL/PRETREATMENT:(organic sediment): acid	/alkali/acid	

NOTE: Five additional samples, FSTC2-RC1, FST3-RC1, -RC2, FST3-RC3 and FST4-RC1, were submitted but not analyzed (as instructed).

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950A.D.). By International convention, the modern reference standard was 95% of the C14 content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C14 half life (5568 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards. Measured C13/C12 ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the C13/C12 value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C14 age.

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables:estimated C13/C12=-25:lab mult.=1)		
Laboratory Number:	Beta-80451	
Conventional radiocarbon age*:	3650 +/- 70 BP	
Calibrated results: (2 sigma, 95% probability)	cal BC 2195 to 1870 and cal BC 1830 to 1780	
* C13/C12 ratio estimated		
Intercept data:		
Intercept of radiocarbon age with calibration curve:	cal BC 1985	
1 sigma calibrated results: (68% probability)	cal BC 2125 to 2065 and cal BC 2060 to 1910	



Talma, A. S. and Vogel, J. C., 1993, Radiocarbon 35(2), p317-322



Stuiver, M., Long, A., Kra, R. S. and Devine, J. M., 1993, Radiocarbon 35(1)

Beta Analytic Radiocarbon Dating Laboratory

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CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-26.2:lab. mult=1)		
Laboratory Number:	Beta-80493	
Conventional radiocarbon age:	1750 +/- 50 BP	
Calibrated results: (2 sigma, 95% probability)	cal AD 160 to 415	
Intercept data:		
Intercepts of radiocarbon age with calibration curve:	cal AD 265 and cal AD 290 and cal AD 320	
1 sigma calibrated results: (68% probability)	cal AD 240 to 380	



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ANALYTICAL PROCEDURES AND FINAL REPORT

RONALD E. HATFIELD Laboratory Manager

CHRISTOPHER PATRICK TERESA A. ZILKO-MILLER Associate Managers

FINAL REPORT

This package includes the final date report, this statement outlining our analytical procedures, a glossary of pretreatment terms, calendar calibration information, billing documents (containing balance/credit information and the number of samples submitted within the yearly discount period), and peripheral items to use with future submittals. The final report includes the individual analysis method, the delivery basis, the material type and the individual pretreatments applied. Please recall any correspondences or communications we may have had regarding sample integrity, size, special considerations or conversions from one analytical technique to another (e.g. radiometric to AMS). The final report has also been sent by fax or e-mail, where available.

PRETREATMENT

Results were obtained on the portion of suitable carbon remaining after any necessary chemical and mechanical pretreatments of the submitted material. Pretreatments were applied, where necessary, to isolate ¹⁴C which may best represent the time event of interest. Individual pretreatments are listed on the report next to each result and are defined in the enclosed glossary. When interpreting the results, it is important to consider the pretreatments. Some samples cannot be fully pretreated making their ¹⁴C ages more subjective than samples which can be fully pretreated. Some materials receive no pretreatments. Please read the pretreatment glossary.

ANALYSIS

Materials measured by the radiometric technique were analyzed by synthesizing sample carbon to benzene (92% C), measuring for ¹⁴C content in a scintillation spectrometer, and then calculating for radiocarbon age. If the Extended Counting Service was used, the ¹⁴C content was measured for a greatly extended period of time. AMS results were derived from reduction of sample carbon to graphite (100 %C), along with standards and backgrounds. The graphite was then sent for ¹⁴C measurement in an accelerator-mass-spectrometer located at one of three collaborating laboratories; Lawrence Livermore National Laboratory (CAMS) in California, Eidgenössische Technische Hochschule University (ETH) in Zürich, or Oxford University (Ox) in Oxford, England.

CALENDAR CALIBRATION

The "Conventional C14 Age (*)" is the result after applying C13/C12 corrections to the measured age and is the most appropriate radiocarbon age (the "*" is discussed at the bottom of the final report). Applicable calendar calibrations are included for organic materials and fresh water carbonates between 0 and 10,000 BP and for marine carbonates between 0 and 8,300 BP. If certain calibrations are not included with this report, the results were either too young, too old, or inappropriate for calibration. It is important to read the calibration explanation sheet before interpreting the results (especially for calcareous materials).

Calibrations of radiocarbon age determinations are applied to convert BP results to calendar years. The short term difference between the two is caused by fluctuations in the heliomagnetic modulation of the galactic cosmic radiation and, recently, large scale burning of fossil fuels and nuclear devices testing. Geomagnetic variations are the probable cause of longer term differences.

The parameters used for the corrections have been obtained through precise analyses of hundreds of samples taken from known-age tree rings of oak, sequoia, and fir up to 7,200 BP. The parameters for older samples, up to 22,000 BP, as well as for all marine samples, have been inferred from other evidence. Calibrations are presently provided for terrestrial samples to about 10,000 BP and marine samples to about 8,300 BP.

The Pretoria Calibration Procedure program has been chosen for these dendrocalibrations. It uses splines through the tree-ring data as calibration curves, which eliminates a large part of the statistical scatter of the actual data points. The spline calibration allows adjustment of the average curve by a quantified closenessof-fit parameter to the measured data points. On the following calibration curves, the solid bars represent one sigma statistics (68% probability) and the hollow bars represent two sigma statistics (95% probability). Marine carbonate samples that have been corrected for $\delta^{13/12}$ C, have also been corrected for both global and local geographic reservoir effects (as published in Radiocarbon, Volume 35, Number 1, 1993) prior to the calibration. Marine carbonates that have not been corrected for $^{-\delta}$ ^{13/12}C, have been adjusted by an assumed value of 0 ‰ in addition to the reservoir corrections. Reservoir corrections for fresh water carbonates are usually unknown and are generally not accounted for in those calibrations. In the absence of measured δ $^{13/12}C$ ratios, a typical value of -5 ‰ was assumed for freshwater carbonates. There are separate calibration data for the Northern and Southern Hemisphere. Variables used in each calibration are listed below the title of each calibration page.

(Caveat: the calibrations assume that the material dated was living for exactly ten or twenty years (e.g. a collection of 10 or 20 individual tree rings taken from the outer portion of a tree that was cut down to produce the sample in the feature dated). For other materials, the maximum and minimum calibrated age ranges given by the computer program are uncertain. The possibility of an "old wood effect" must also be considered, as well as the potential inclusion of some younger material in the total sample. Since the vast majority of samples dated probably will not fulfill the ten/twenty-year-criterium and, in addition, an old wood effect or young carbon inclusion might not be excludable, these dendrocalibration results should be used only for illustrative purposes. In the case of carbonates, reservoir correction is theoretical and the local variations are real, highly variable and dependant on provenience. The age ranges and, especially, the intercept ages generated by the program must be considered as approximations.)

EXPLANATION OF THE BETA ANALYTIC DENDRO-CALIBRATION PRINTOUT



CALIBRATION OF RADICARBON AGE TO CALENDAR YEARS

References:

Vogel, J. C., Fuls, A., Visser, E. and Becker, B., 1993, Radiocarbon 33(1), p73-86 Talma, A. S. and Vogel, J. C., 1993, Radiocarbon 35(2), p317-322 Stuiver, M., Long, A., Kra, R. S. and Devine, J. M., 1993, Radiocarbon 35(1)

Results prepared by: -

Beta Analytic, Inc., 4985 S.W. 74th Court, Miami, Florida 33155

Reporting results (recommended):

- 1. List the radiocarbon age with its associated 1 sigma standard deviation in a table and designate it as such.
- 2. Discussion of ages in the text should focus on the 2 sigma calibrated range.

Derivation of a radiometric or accelerator dendro-calibrated (CALENDAR) date requires use of a <u>CONVENTIONAL</u> radiocarbon date (Stuiver and Polach)¹. The conventional date is a basic radiocarbon date that has been normalized to the modern standard through the use of C13/C12 ratios* (analyzed or estimated). The statistical error (+/-) on an <u>analyzed</u> C13/C12 value is quite small and does not contribute significantly to the combined error on the date. However, use of an <u>estimated</u> C13/C12 ratio for an unknown sample may incur a very large combined error term. This is clearly illustrated in the figure below (Gupta & Polach; modified by J. Head)² where the possible range of C13/C12 values for a particular material type may be so large as to preclude any practical application or correction.

In cases where analyzed C13/C12 values are not available, we have provided (for illustration) dendro-calibrations assuming a mean "chart" value, but <u>without</u> an estimated error term.

Where a sample carbon reservoir different from the modern oxalic acid/wood modern standard (e.g. shell) is involved, a further correction must be employed; the necessary variables are displayed on the calibration sheet.



¹Stuiver, M. and Polach, H.A., 1977. Discussion: Reporting of 14-C data, Radiocarbon, 19, 355-363.

²Gupta S.K. and Polach H.A., 1985. Radiocarbon dating practices at ANU Handbook,p.114. Radiocarbon Laboratory, Research School of Pacific Studies, ANU, Canberra.

*Radiocarbon is incorporated into various materials by different pathways and this introduces differing degrees of <u>isotopic fractionation</u>. The C13/C12 ratio of any material is the millesimal difference of the sample to the carbonate PDB standard and is directly related to the C14/C12 ratio. The degree of sample C-14 enrichment or depletion then is normalized to that of the modern standard.

PRETREATMENT GLOSSARY

Pretreatment of submitted materials is required to eliminate secondary carbon components. These components, if not eliminated, could result in a radiocarbon date which is too young or too old. Pretreatment does not ensure that the radiocarbon date will represent the time event of interest. This is determined by the sample integrity. The old wood effect, burned intrusive roots, bioturbation, secondary deposition, secondary biogenic activity incorporating recent carbon (bacteria) and the analysis of multiple components of differing age are just some examples of potential problems. The pretreatment philosophy is to reduce the sample to a single component, where possible, to minimize the added subjectivity associated with these types of problems.

"acid/alkali/acid"

The sample was first gently crushed/dispersed in deionized water. It was then given hot HCI acid washes to eliminate carbonates and alkali washes (NaOH) to remove secondary organic acids. The alkali washes were followed by a final acid rinse to neutralize the solution prior to drying. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of the sample. Each chemical solution was neutralized prior to application of the next. During these serial rinses, mechanical contaminants such as associated sediments and rootlets were eliminated. This type of pretreatment is considered a "full pretreatment".

Typically applied to: charcoal, wood, some peats, some sediments, textiles

"acid washes"

Surface area was increased as much a possible. Solid chunks were crushed, fibrous materials were shredded, and sediments were dispersed. Acid (HCI) was applied repeatedly to ensure the absence of carbonates. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of each sample. The sample, for a number of reasons, could not be subjected to alkali washes to ensure the absence of secondary organic acids. The most common reason is that the primary carbon is soluble in the alkali. Dating results reflect the total organic content of the analyzed material. Their accuracy depends on the researcher's ability to subjectively eliminate potential contaminants based on contextual facts.

Typically applied to: organic sediments, some peats, small wood or charcoal, special cases

"collagen extraction"

The material was first tested for friability ("softness"). Very soft bone material is an indication of the potential absence of the collagen fraction (basal bone protein acting as a "reinforcing agent" within the crystalline apatite structure). It was then washed in deionized water and gently crushed. Dilute, cold HCl acid was repeatedly applied and replenished until the mineral fraction (bone apatite) was eliminated. The collagen was then dissected and inspected for rootlets. Any rootlets present were also removed when replenishing the acid solutions. Where possible, usually dependant on the amount of collagen available, alkali (NaOH) was also applied to ensure the absence of secondary organic acids.

Typically applied to: bones

"acid etch"

The calcareous material was first washed in de-ionized water, removing associated organic sediments and debris (where present). The material was then crushed/dispersed and repeatedly subjected to HCl etches to eliminate secondary carbonate components. In the case of thick shells, the surfaces were physically abraded prior to etching down to a hard, primary core remained. In the case of porous carbonate nodules and caliche, very long exposure times were applied to allow infiltration of the acid. Acid exposure times, concentrations, and number of repetitions, were applied accordingly with the uniqueness of the sample.

Typically applied to: shells, caliche, calcareous nodules

"neutralized"

Carbonates precipitated from ground water are usually submitted in an alkaline condition (ammonium hydroxide or sodium hydroxide solution). Typically this solution is neutralized in the original sample container, using deionized water. If larger volume dilution was required, the precipitate and solution were transferred to a sealed separatory flask and rinsed to neutrality. Exposure to atmosphere was minimal.

Typically applied to: Strontium carbonate, Barium carbonate (i.e. precipitated ground water samples)

"none"

No laboratory pretreatments were applied. Special requests and pre-laboratory pretreatment usually accounts for this. This would never be the circumstance without the knowledge of the submitter.