# 4.1 COMPARISON OF APPROACHES TO DATING ATLANTIC COASTAL PLAIN SEDIMENTS, VIRGINIA BEACH, VIRGINIA

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## INTRODUCTION

In this study we characterized and dated a stratified section of middle to late Pleistocene strata exposed in a borrow pit in Virginia Beach, Virginia (Fig. 4.1-1). We collected sediment, coral, and mollusk samples for the intercomparison of infrared stimulated luminescence (IRSL), uranium-series (U-series), and amino acid racemization (AAR) geochronologic methods. This intercomparison is important for independently testing and validating these methods for dating middle and late Pleistocene sediments of the Atlantic Coastal Plain.

The borrow pit site locally is known as Gomez Pit, owned and operated by the E. V. Williams Co. of Virginia Beach. It is one of several borrow pits that have yielded a large amount of Quaternary litho- and chrono-stratigraphic information for the region the last 30 years. A recent paper by Mirecki and others (1995) summarizes the geochronologic framework for the current intercomparison study, and Wehmiller and others (1989) provide an overview of relevant literature and the geomorphology, stratigraphy, and geochronology of Gomez Pit and nearby sites. Other recent publications that discuss the geochronology at Gomez Pit in a broader regional context include Groot and others (1990), Toscano and York (1992), and Wehmiller and others (1992; 1995). The site was chosen for this intercomparison study because of the extensive background knowledge that was already available. Because Gomez Pit is an active borrow pit, the exposures are always changing and we were fortunate in having access to some fresh outcrops (less than one year of exposure) for sampling during the present study.

New geochronologic results are presented here for IRSL dating of host sediments, Useries dating of corals, and AAR dating of mollusks, obtained on samples collected from Gomez Pit in August and September, 1995. Samples from previously obtained collections at Gomez Pit were also analyzed. The spacing and timing of the collections of different outcrops at a single site is an important part of the interpretation of results as weathering and diagenesis can play important roles in AAR dating.



Figure 4.1-1. Regional map of the mid-Atlantic coastal plain and expanded view of the area of southeastern Virginia and northeastern North Carolina. See Mirecki and others (1995) (see Appendix I and II for further locality information). GP= Gomez Pit; Mck = Moyock; NL = New Light Pit; YP = Yadkin Pit.

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## FIELD STUDY AND SAMPLING EFFORT

The perennially changing working faces of Gomez Pit have been visited and sampled over the past 15 years by geologists from numerous institutions (Fig. 4.1-2). Previous investigations involved aspects of sedimentologic, paleoenvironmental reconstruction and/or geochronology. Recent collections of geochronologic data were made by Mirecki (1990) and Wehmiller (unpublished data). Sites used by Mirecki for her Ph.D. research (1982-1987;





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Mirecki, 1990) were sampled as the face was excavated. Coral and sediment samples were collected for the purposes of U-series and luminescence dating by Wehmiller in 1988 and 1989 at some of these sites (Fig. 4.1-2). For this study, exposures in the southeastern portion of the mapped area (Fig. 4.1-2) were sampled extensively in August and September, 1995. Two measured sections (MS#1 and MS#2) in the newer portions of the "new pit" were studied in September, 1995, and a third section (MS#3), in the older part of the new pit, was stratigraphically tied to these measured sections by walking out all of the lithologic contacts. Seven of the nine luminescence samples were taken from MS#1, MS#2, and MS#3 in August, 1995.

The 1995 sampling program was initiated specifically for this project. In June, 1995, we visited Gomez Pit for one day to determine the feasibility of conducting the research. Plans were made to collect samples for dating. Samples for luminescence dating were acquired on August 17-19, 1995. We also collected some mollusk samples at this time, being particularly interested to collect shells with clear stratigraphic relation to the IRSL samples. Photographic documentation of collection sites was also conducted.

After the August collection trip, plans were made for a return to Gomez Pit for additional sampling and a "field conference" involving all interested parties. This work was conducted between September 22 and 25, 1995. Mollusk sampling, surveying, general mapping, and stratigraphic section description were conducted, along with additional photography. Gerald Johnson, of the Geology Department at the College of William and Mary, along with a large group from the University of Delaware (Delaware), participated in this September field work. The U-series geochronologist in this project, Ken Ludwig of the U.S. Geological Survey (USGS) Denver, also was not able to participate in this September visit. Because of Ludwig's time constraints, it was agreed that the U-series results that would be incorporated into this study would be from samples previously collected by J. Wehmiller and recently (1994-1995) analyzed by Ludwig in the context of some independent collaborative research between Delaware and the USGS.

## STRATIGRAPHIC DESCRIPTION OF THE UNITS EXPOSED IN GOMEZ PIT

The nomenclature and interpretation of the general stratigraphic sequence at Gomez Pit is summarized in Mirecki and others (1995) and illustrated in Figures 4.1-3 and -4, from Mixon and others (1982) and Peebles and others (1984), respectively. Measured sections MS#1, MS#2, and MS#3 (Figs. 4.1-5, -6 and -7) provide further lithostratigraphic detail of the section exposed at Gomez Pit and show the positions of several of the luminescence and mollusk samples collected in this study. The emphasis in our sampling was to obtain geochronologic samples from the "upper" or serpulid unit and the "lower" or oyster biostrome unit (see Fig. 4.1-4) because previous aminostratigraphic results (Mirecki and others, 1995) indicate that there is a substantial difference in age between these two units. Mollusks and luminescence sample GP10 were also collected from the Chowan River Formation, which is exposed in trenches near the center of the pit. Corals were sampled from the serpulid unit only.









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Figure 4.1-4 Stratigraphic diagram for Gomez Pit, from Peebles and others (1984).

## SAMPLES AND ANALYTICAL METHODS

Geochronologic sample numbers and data for this study are presented in Tables 4.1-1 (IRSL), -2 (U-series) and -3 (AAR). Samples locations are shown in Figures 4.1-2 and 4.1-8. Sample preparation and analytical methods followed normal practices employed in each of the participating laboratories, and are described briefly below. More information on these dating methods, including principles and methodology, is presented by Forman and others (this volume) on luminescence geochronology, Ku (this volume) on U-series, and Wehmiller and Miller (this volume) on AAR.

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Figure 4.1-5 Measured section #1, Sept. 1995. See Figure 4.1-2 for location.



Figure 4.1-6. Measured section #2, Sept. 1995. See Figure 4.1-2 for location.



Figure 4.1-7. Measured section #3, Sept. 1995. No detailed description done for this section, but survey markers (blue and pink flags) can be used to match this section to sections #1 and #2.



Gomez surveys and TL samples

Figure 4.1-8 Surveys of marker beds in sections identified in Figure 4.1-2, including locations of measured sections #1-3. Sections are projected onto line A-A' as shown in Figure 4.1-2.

#### Luminescence Geochronology

Luminescence analysis was carried out at the University of Quebec in Montreal under the direction of M. Lamothe, primarily on samples of the sand beds, except two samples of mud (GP6 and GP8). From the sands, the 150-200  $\mu$ m size fraction of feldspar was separated using sieve and densimetric methods. Sample GP7 yielded little of this grain size and a second split of 300-500  $\mu$ m size fraction was prepared. GP6 and GP8 samples are fine-grained mud and the 4-8  $\mu$ m size fraction was selected for analysis. Although no mineralogical separation was possible on these samples, it is expected that they contain feldspar which is sensitive to IRSL (880 nm photons).

Multiple aliquots of sample were mounted on aluminum planchets (if sand) or discs (if fine silt). All samples were subsequently illuminated using an 880 nm infrared source. This is done for normalization as well as for luminescence stimulation to derive the growth of the luminescence signal upon dosing. The choice of this stimulation wavelength is based on a resonance effect for feldspar as shown by Hutt and Jaek (1989) and Spooner (1993).

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TABLE 4.1-1.	LUMINESCENCE SAMPLES FROM GOMEZ PIT
	VIRGINIA BEACH, VIRGINIA

Sample	Location	
	Sampled by J.Wehmiller, May 1989, from site $06076(B)^1$	
VIR-1	ca. 50 cm below oyster bed	
VIR-2	ca. 20 cm above oyster bed, below Mulina bed	
VIR-3	Serpulid bed, lower part	
VIR-4	Serpulid bed, upper part	
	Sampled by Lamothe and Wehmiller, August 1995	
GP1	Upper sand unit, MS#1	
GP2	Above serpulid bed, below blue flag, MS#3	
GP3	Within serpulid bed, MS#3	
GP4	Below serpulid bed, below pink flag, MS#3	
GP5	Below oyster bed, MS#2	
GP6/7	Below oyster bed, MS#3	
GP8	Below oyster bed	
GP9	No sample <sup>2</sup>	
GP10	Chowan River <sup>3</sup>	
GP0	Beach sand, Cape Henry <sup>4</sup>	

<sup>1</sup>VIR-1 through 4 were collected by "coring" into a freshly exposed surface on the outcrop with a 2-lb coffee can. This sampling was done while the outcrop was in shadow and the actual sampling position was further shaded with a black plastic garbage bag. These samples were taken with no specific plan for luminescence analyses, but were later (1991) sent to M. Lamothe for some preliminary analysis.
 <sup>2</sup>Number assigned for sample that would be taken in time permitted.
 <sup>3</sup>Sampled to determine "infinite" TL signal for site, and to evaluate local fading effects.

<sup>4</sup>Sampled to determine "time zero" TL signal for local sediments.

## TABLE 4.1-2. CORAL SAMPLES ANALYZED FOR U-SERIES ISOTOPE GEOCHRONOLOGY

Identification
Sampled by J.Wehmiller, July 1989, from site 06076 serpulid bed <sup>1</sup>
Septastrea <sup>2</sup>
Astrangia
SeptastreaV

<sup>1</sup>Analyzed by Ludwig at USGS, 1994-1995.

<sup>2</sup>Coral specimens (*Septastrea*) from a nearby site (Moyock, North Carolina - see Fig. 4.1-1) are also reported here because these samples were analyzed at the same time and because they were part of the development of a sample preparation scheme that was employed on the Gomez samples. Number assigned for sample that would be taken in time permitted.

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TABLE 4.1-3.	MOLLUSK SAMPLES ANALYZED FOR AMINO ACID RACEMIZATION	V
	AS PART OF THIS STUDY	

Sample	Identification	Date Collected
	Gomez Pit Collections	
JW95-035	Mercenaria within oyster bed at MS#3	8/95
JW95-037	Mercenaria within oyster bed at GP8	8/95
JW95-038	Mercenaria within Chowan River Fm. at GP10	8/95
JW95-043	Mercenaria within serpulid zone, MS#2	9/95
<b>JW</b> 95-044	Mercenaria within serpulid zone, MS#3	9/95
JW95-046	Mercenaria within upper part of oyster bed, MS#2	<del>9</del> /95
JW95-048	Mercenaria within lower part of oyster bed,	9/95
	50 m northeast of MS#2	
	Earlier collections: serpulid zone, Mercenaria samples, C	Gomez Pit
JW89-104	Locality 06076A	
JW89-106	Locality 06076A	
JW89-121	Locality 06076A	
JW89-133	Locality 06076A	
JW89-139	Locality 06076A	
JW89-141	Locality 06076A	
JW89-148	Locality 06076A	
GP278	Locality 06056	
	Earlier collections: oyster bed Mercenaria sample	es
JW88-46	Locality 06074	
JW89-162	Locality 06076A	
JW89-163	Locality 06076A	
JW89-170	Locality 06076B	
JW89-173	Locality 06076B	
	Moyock, North Carolina <sup>1</sup>	
JW93-61-1, -	-2, -3, -4	

<sup>1</sup>Analyzed during this study for intrashell comparison and evaluation of internal consistency.

#### **U-Series Geochronology**

The analysis of U-series isotopic systematics followed standard laboratory procedures employed by the USGS. However, the actual physical preparation of the coral samples was different from that employed in previous coral U-series studies of Atlantic coastal plain sites (Szabo, 1985). Because the thermal ionization mass spectrometry (TIMS) U-series method permits analysis of carbonate samples weighing as little as 100 mg, it was possible to clean large (ca. 3000 mg) coral samples to the most robust (least contaminated) fragments prior to analysis. This strategy has now been employed on approximately 10 coastal plain samples (J. Wehmiller,

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unpublished data) including the samples listed in Table 4.1-2. The actual sample reduction, which has been done at the University of Delaware under the direction of J. Wehmiller, consists of drilling and grinding of the coral while it is held in a dish of distilled water and viewed under a binocular microscope. Tungsten carbide dental drill tips (generally < 0.25 mm) are used for this purpose. As drilling proceeds, coral samples disintegrate leaving a residue of polyp wall material (fragments usually weighing about 10 mg each) and pulverized material that remains in suspension. The TIMS U-series analysis is usually done on a collection of about 15 of these individual fragments. The entire mechanical sample reduction process usually requires about 10 hours for each sample.

#### AAR Geochronologic Methods

The amino acid racemization analyses reported here involved both gas chromatographic (GC) and high-pressure liquid chromatographic (HPLC) methods routinely reported in the literature (Wehmiller and Miller, this volume). As part of current research at Delaware, we are making a concerted effort to analyze the same sample extract by both of these procedures, rather than doing separate analyses of a single shell by different methods at different times. Consequently, we are able to report here both GC and HPLC results obtained simultaneously on a specific sample hydrolyzate. Additionally, because of ongoing research at Delaware, we have been analyzing (by both GC and HPLC) individual layers of *Mercenaria* (see Fig. 4.1-9) to





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evaluate possible shell structural effects on racemization (because of intrinsic mineral/organic effects on racemization kinetics or extrinsic differences in diagenetic alteration of different shell layers) and to use these intra-shell differences in apparent racemization as aminostratigraphic tools. Some, but not all, of the shells analyzed here were sampled for intra-shell analysis. Interlaboratory comparison samples (Wehmiller, 1984) were analyzed routinely (at least one ILC sample with each batch of ten shells) during the course of the amino acid analyses reported here.

## **DISCUSSION OF RESULTS**

#### Apparent IRSL Age Estimates

Luminescence geochronology on Gomez Pit samples yielded age estimates that are significantly younger than expected and as determined by U-series and AAR methods. The dosimetric and luminescence data are shown on Tables 4.1-4, -5, and -6. The histogram from the single grain analyses, after prompt measurement and a waiting period, are shown on Figure 4.1-10. This type of test is designed to assess the datability of a sample (Lamothe and Auclair, 1997). Growth curves are presented in Figure 4.1-11. As presented in Table 4.1-6, reported age estimates range from 32 to 87 ka, if only the prompt measurements are considered. Even the GP10 sample, from the Pliocene Yorktown Formation yielded an anomalously young age of 342 ka. Age estimates for the lower and upper Norfolk Formation cluster at around 70-90 ka and 30-40 ka, respectively. These results indeed confirm that there are at least two high sea level stands represented at the Gomez Pit. A stage 7 correlation is suggested for sample VIR-1, although its ca. 160 ka age is a minimum.

The distribution of  $R_I$  values for sample GP3 (Fig. 4.1-10a) and the fact that the growth curve data are mostly reproducible (lower reproducibility for GP7 and GP10), are a clear indication that the samples were properly bleached before deposition. In that sense, the set of samples from Gomez Pit constitutes an ideal group of samples to study the second postulate in luminescence geochronology: the stability of luminescence for the constituent feldspar grains. Inasmuch as the geochronologic control at Gomez Pit is not well established, one can nevertheless recognize that the ages reported here are too young on the basis of paleo-sea level reconstruction. Wehmiller and others (1989) have shown that the periods of high sea level in the area correspond to unique isotopic stages, i.e., the ones that represent periods characterized by a return of oxygen isotopic ratios close to the modern value. These periods are isotopic stages 5a (according to Ludwig and others, 1996), 5e, 7, 9 and so on. None of these periods are younger than ca. 75 ka so that it can confidently be assessed that the IRSL ages are too young. The processes through which underestimation of ages can result is discussed below.

As discussed above, the second assumption in luminescence geochronology is that the electrons in the dated traps are bound tightly at electron sites. It has been known for several years that at least feldspar from volcanic terranes is prone to anomalous fading, which is defined here as the loss of luminescence signal with time from presumably stable deep traps (Wintle, 1973). Even though Wintle and Huntley (1982) had required that anomalous fading tests be carried out in any dating program, the advent of preheating in the laboratory procedures has had the malign effect of reducing the possibility of monitoring such effect since most of the component that is unstable over the period of observation in the laboratory is eradicated by the thermal treatment. Recently, Spooner (1993) demonstrated that specimens of feldspar minerals

Sample	Fraction	U (ppm)	Th (ppm)	K (percent)	Water content in situ	Laboratory saturation
GP1	wc	0.2	0.4	0.82	0.030	0.301
GP1	kut A	0.3	0.5	0.51	0.013	
GP1	kut B	0.6	1.2	0.73	0.063	
GP1	feldspath	0.1	<0.1	13.16		
GP2	wc	0.6	2.0	1.10	0.039	0.300
GP2	kut A	0.8	1.8	1.09	0.067	
GP2	kut B	0.7	1.4	1.28	0.043	
GP2	feldspath	0.1	0.2	13.95		
GP3	wc	1.2	1.6	1.12	0.116	0.136
GP3	kut A	1.0	1.2	0.94	0.049	
GP3	kut B	1.4	2.1	1.36	0.052	
GP3	feldspath	0.4	0.2	13.78		
GP4	wc	0.8	2.0	0.48	0.046	0.287
GP4	kut A	0.8	1.6	0.56	0.029	
GP4	kut B	0.7	1.7	0.43	0.029	
GP4	feldspath	0.2	0.4	13.37		
GP5	wc	1.2	2.0	0.75	0.208	0.240
GP5	kut A	1.8	3.4	0.88	0.210	
GP5	kut B	1.8	3.1	0.75	0.174	
GP5	feldspath	1.4	1.6	14.86		
GP6	wc	4.0	8.7	1.95	0.521	0.694
GP7	wc	0.4	0.6	0.45	0.244	
GP7	kut A	1.9	5.6	1.27		0.360
GP7	kut B	1.7	3.7	1.03	0.100	
GP7	feldspath	<0.1	1.1	14.54		
GP8	wc	2.9	8.9	2.09	0.608	0.650
GP8	kut	4.2	12.6	2.30		
<b>GP10</b>	wc	1.3	2.3	0.84		
GP10	feldspath	0.4	0.8	12.12		

TABLE 4.1-4. GEOCHEMISTRY AND WATER CONTENT OF THE IRSL SAMPLES

K, U and Th analyzed by instrumental neutron activation (INAA)

Water content = water mass/dry mass

from museum collections were prone to fading, whatever their composition. In this Ph.D. study, he monitored the luminescence signal from several types of feldspar over time periods in excess of one year and most of his specimens did fade. As a follow up of this study, the monitoring of several feldspar grains from different geological contexts and ages has been carried out in our laboratory. In most of the cases, there were several grains in the samples investigated that were shown to lose some 10-20 percent of their luminescence signal over time periods of less than 2 months. Moreover, the aliquots used for the construction of the growth curves also have been

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Sample	Grain size	Alpha dose (Gy/ka)	External Beta dose (Gy/ka)	Internal Beta dose (Gy/ka)	Gamma dose (Gy/ka)	Dose rate (Gy/ka)
GP1	150-250 μm		0.535	0.752	0.207	1.644
GP2	150-250 μm		0.778	0.797	0.373	2.098
GP3	150-250 µm		0.885	0.787	0.430	2.252
GP4	150-250 µm		0.416	0.764	0.252	1.582
GP5	150-250 μm		0.588	0.849	0.408	1.995
GP6	4-8 μm	0.518	1.298		0.771	2.737
GP7	300-500 µm		0.257	1.659	0.385	2.451
GP8	4-8 μm	0.446	1.314		0.860	2.770
GP10	150-250 μm		0.583	0.693	0.330	1.756

TABLE 4.1-5.	DOSE	RATE	DATA
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Notes 1 - a value for samples GP6 and GP8 = 0.06

2- External Beta dose corrected for attenuation, Internal Bata dose rate corrected for absorption

3- Cosmic dose estimated at 0.15 Gy/ka

## TABLE 4.1-6. APPARENT IRSL AGES OF GOMEZ PIT SAMPLES

Sample	Dose rate (Gy/ka)	Paleodose (Gy)	IRSL age (ka)
VIR 1	1.89	$73 \pm 4$	38.6
VIR 2	2.76	95 ± 5	34.4
VIR 3	1.61	259 ± 23	161

Sample	Dose rate		Paleodoses (C	Gy)	IRSL ages (ka)			
	(Gy/ka)	De (0)	De (1)	De (2)	Age (0)	Age (1)	Age (2)	
GP1	1.64	58.4 ± 1.9	63.9 ± 1.4	64.1 ± 2.5	35.5	38.9	39.0	
GP2	2.10	$70.4 \pm 2.7$		$90.3 \pm 4.2$	33.6		43.0	
GP3	2.25	$71.7 \pm 3.2$	77.7 ± 3.4	$85.8 \pm 3.4$	31.8	34.5	38.1	
GP4	1.58	$65.1 \pm 2.4$		$68.6 \pm 2.7$	41.2		43.4	
GP5	1.99	$142 \pm 11$		175 ± 6	71.2		87.7	
GP6	2.74	167 ± 10	*	$195 \pm 10$	61.0		71.2	
GP7	2.45	$213 \pm 20$	$231 \pm 49$	$239 \pm 25$	86.9	94.3	97.5	
GP8	2.77	193 ± 19		237 ± 19	69.7		85.6	
GP10	1.76	$600 \pm 55$	826 ± 70		342	470		

Notes De (0) = prompt measurement; De (1) and De (2) = delayed measurements De (1) : delay = 45 to 50 days, except for GP10 where delay = 25 days De (2) : delay = 127 to 137 days Error for age around 10 percent

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Figure 4.1-10. Histograms of  $R_1$  for GP3 single grains after prompt measurement (a), and delays after 8 and 34 days (b and c, respectively).

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Figure 4.1-11. Growth curves for for samples GP1, GP3, GP6 and GP10, after prompt and delayed measurements (a, b, c, and d, respectively).

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remeasured some time after irradiation and these showed a decrease of signal, particularly for those with high doses.

For several samples from Gomez Pit, the same delayed measurements were performed after periods of between 25 to 137 days. They all showed a decrease of luminescence signal. The same is observed for the  $R_I$  values measured from single grains (Fig. 4.1-10b) over a period of 34 days. Such samples and methods are therefore not suited for dating the depositional age for these samples. Consequently, these estimates are considered to be minimum ages.

#### Anomalous Fading in Feldspar

The stability of electrons trapped in defects of feldspathic minerals is a matter of strong debate. Ever since Wintle (1973) discovered the problem of anomalous fading, this malign behavior of feldspars has escaped thorough understanding of its fundamental physical basis. In brief, electrons trapped in minerals are believed to be tightly preserved from untrapping because of the need for an external energy source to excite them (see Forman and others, this volume). Upon glowing a sample, heat can provide enough energy to excite the electron so that it may escape from the trap, go through the conduction band, and undergo recombination, some of which is radiative and leads to thermoluminescence. Optical stimulation from, for example, 2 eV photons can provide enough energy to the electron so that it may escape and recombine. However it is known that, provided a small amount of thermal energy (a few tens of °C), recombination can take place through the so-called localized transitions (Templer, 1986) without the need for the electron to reach the conduction band (Fig. 4.1-12). On the other hand,







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theoretical calculation suggests that a crystal held at room temperature should keep constant its trapped electron density. Nevertheless, Spooner (1993) has demonstrated that even at 10°C, some electrons can escape the dated trap through athermal tunneling, a phenomenon for which an explanation would go beyond the scope of this report. The main point here is that, after some time, there could be a decrease in electron density in any trap that suffers from anomalous fading. This process takes some time and can be unnoticed by the TL/OSL practitioner in routine dating procedures.

In the case of the Gomez Pit samples, the first test for stability was the remeasurement of the aliquots used for IRSL dating a few weeks to a few months after the prompt measurements. As shown on Figure 4.1-11, there is a significant decrease in luminescence of the irradiated aliquots compared to the natural ones. The latter, by definition, is a stable luminescence signal. A second experiment was based on the monitoring of individual grains at different times after dosing. Again, for some grains, the decrease was severe, and it demonstrated that the samples are badly fading so that the ages should be considered as minimal.

#### **U-series** Results

U-series results presented here (Table 4.1-7) confirm previous U-series results for samples from the serpulid unit of Gomez Pit (Szabo, 1985). All previous U-series results were obtained by the alpha-spectrometric method. New data presented here provide the first application of the TIMS method to U.S. Atlantic coastal plain corals. Alpha-spectrometry age estimates of between 70 and 80 ka (substage 5a) were obtained on several coral samples from Gomez, Moyock, and other nearby borrow pits in the region. Although the new results do not change any of the conclusions related to the previous results, they do constitute a significant "challenge" to oxygen-isotope-based sea-level curves, which would predict sea levels during substage 5a to be 10 m or more lower than the current elevation of the serpulid unit. The

Sample	U, ppm	230Th/232Th	<sup>230</sup> Th/U age, ky	Age error, ky	<sup>234</sup> U/ <sup>238</sup> U, initial	Initial error
JW89-117 <sup>1</sup>	2.3	98	79.8	1.0	1.150	0.02
JW89-118 <sup>1</sup>	2.1	430	74.2	0.9	1.142	0.02
JW89-124B <sup>1</sup>	1.9	236	75.8	6.9	1.140	0.04
JW92-48D <sup>2</sup>	2.6	11	78.7	2.8	1.154	0.07
JW92-48C <sup>2</sup>	2.4	98	79.8	1.0	1.150	0.02

## TABLE 4.1-7. GOMEZ PIT U-SERIES TIMS DATA

<sup>1</sup> Calcite/aragonite determinations have been made on these samples; there is no more than a "trace" of calcite (<3 percent).

<sup>2</sup> Samples JW92-48D and C are "dirty" and "clean" splits of the same coral sample, from Moyock, North Carolina. The cleaned sample was prepared in the drilling/reduction manner described in the text, as were the three Gomez samples (89-117, 89-118, and 89-124B). Note that the cleaning of JW92-48 reduced the <sup>232</sup>Th content and improved the age and initial <sup>234</sup>U/<sup>238</sup>U errors.

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serpulid unit was deposited below mean sea level. The implications of these substage 5a age estimates for coastal plain corals have been debated extensively over the past 15 years (Cronin and others, 1981; Szabo, 1985; Wehmiller and others, 1988) and are the subject of continuing investigation. Some possible diagenetic issues related to these dates will be discussed below. We conclude also that the cleaning procedures used (mechanical reduction to eliminate the most porous portions of the corals) and the use of TIMS technology do improve the analytical results (evidenced in the reduction in <sup>232</sup>Th and the improved precision) but they did not significantly vary from the original age estimate.

### AAR Results

As with previous AAR results (Mirecki, 1990, 1995; J. Wehmiller, unpublished data), the Gomez Pit section consists of two distinct aminozones. Figure 4.1-13 shows the D/L leucine and D/L alanine values for *Mercenaria* samples from previous collections (1985-1989) in the



Figure 4.1-13. D/L leucine and D/L alanine values for *Mercenaria* samples from previously-collected Gomez pit sites (aminozones IIa and IIc - see appendices I and II), from Moyock, North Carolina, and from Holocene samples (<sup>14</sup>C-dated) collected at Corolla, North Carolina (Fig.4.1-1).

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serpulid and oyster units of Gomez Pit. The aminostratigraphic data from these two units cluster into two aminozones "IIa" and "IIc" that are recognized regionally (Wehmiller and others, 1988). Mirecki and others (1995) show the distribution of these aminozones based on Dalloisoleucine/L-isoleucine data obtained by HPLC, with mean A/I values for these two aminozones of 0.16 and 0.32, respectively. In the present study we have employed not only A/I values (obtained by both GC and HPLC) but D/L values for at least four other amino acids (alanine, valine, leucine, and aspartic acid). Because of redesign of the HPLC detection system and modification of detection reagent concentrations, A/I values currently are about 10 percent greater than those obtained on our HPLC between 1985 and 1990 (including all those obtained by Mirecki). Figure 4.1-13 shows that leucine D/L values in "IIa shells" range between 0.22 and 0.32 and alanine D/L values range between 0.38 and 0.50. The lower D/L values are usually (but not always) observed in fragments cut from the edges of the shells (hinge or growth edge. as well as the middle layer in the central portion of the shell), while the higher D/L values in any aminozone are almost always obtained from fragments cut from the inner homogeneous layer (see Fig. 4.1-9 for shell sampling positions and structural terminology). The leucine and alanine values cluster around 0.42 and 0.65, respectively, for aminozone IIc shells.

Leucine and alanine D/L values for *Mercenaria* from Moyock, NC, overlap those for the Gomez IIa aminozone. This nearby site (Fig. 4.1-2) has yielded U-series data overlapping those from Gomez Pit. Figure 4.1-13 also shows leucine and alanine D/L values for Holocene (1<sup>4</sup>C-dated) *Mercenaria* collected at Corolla, NC (Wehmiller and others, 1995). These data help define the regional Holocene aminozone.

Newly obtained data from locations MS#2 (95-043), MS#3 (95-044), GP6/7 (95-035), GP8 (95-037), and GP10 (95-038), and samples 95-046 and 95-048, overlap ranges for the IIa and IIc aminozones from previous work (Figs. 4.1-13 and -14). These results represent *Mercenaria* collected from upper part of the oyster unit in the immediate vicinity of MS#2 (95-046) and *Mercenaria* collected from the lower oyster unit 50 m northeast of MS#2 (95-048). Figure 4.1-14 shows that 95-035 and 95-037 fall within the range of IIc shells from previous collections, confirming the aminostratigraphic correlation that was expected at the time of collection. 95-038 shells plot at or near equilibrium D/L values, also as expected from the known age of these shells (Pliocene/early Pleistocene Chowan River Fm). Sample 95-048 appears to plot with higher D/L values than those from IIc or 95-035 and 95-037, suggesting a greater age for the 95-048 shells. 95-046 shells spread over a wide range of D/L values that barely overlap with the lowest part of the 95-048 range (Fig. 4.1-19). 95-046 shells actual range so widely that they overlap with some of the 95-043 and 95-044 shells, all of which were collected from within the serpulid unit and were therefore expected to have IIa aminozone D/L values.

Results for samples 95-043 and 95-044 are enigmatic. Their A/I and D/L leucine values are generally higher than the same ratios in serpulid unit shells collected from elsewhere in the pit (Appendix II). The serpulid unit shells from Gomez Pit have yielded a large range of A/I and leucine D/L values that was also observed by Mirecki (1990). This range can usually be explained in terms of shell preservation characteristics. Furthermore, it appears that most (if not all) of this range can be duplicated in a single shell by analysis of single shell layers. This intra-shell variation appears to diminish in relative significance as the shells become more racemized.



Gomez Pit Aminozones

Figure 4.1-14. Gomez Pit Aminozones: Leucine and Alanine D/L values from the IIa, IIc, Moyock and Holocene aminozones (Fig. 4.1-10) compared with those from MS#2 (95-043, 95-046, and 95-048), MS#2 (95-044 and 95-035), GP8 (95-037), and GP10 (95-038). Similar clusters of D/L values have been observed by Mirecki (1990).

Belknap (1979) and Wehmiller and Belknap (1982) also noted these ranges of D/L values for the different units, but the number of analyses obtained for the earlier studies was small enough to preclude any firm conclusions regarding diagenetic effects.

The results for 95-046 and 95-048 are important because of their value in understanding the stratigraphy, taphonomy, and geochemical diagenetic processes of the oyster unit that is so prominent throughout Gomez Pit. Previous aminostratigraphic results for *Mercenaria* from the oyster unit have been confusing, with two aminozones being inferred, one representing reworked shells (Mirecki, 1990; Mirecki and others, 1995). The new results for 95-046 and 95-048 reinforce these original conclusions. Furthermore, occasional *Mercenaria* samples from the uppermost part of the oyster unit have yielded aminozone IIa values, indicating that shells of this age burrowed into the underlying oyster unit during the time (i.e., IIa time) of deposition of the overlying sand and serpulid units. Some of the shells from the top of the oyster unit (95-046) also confirm this observation, and at least one of these shells was noted as probably being of this young age at the time of its collection. The consistently higher D/L values for the 95-048

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samples, collected from lower in the oyster unit (see MS#1 and MS#2 descriptions; 95-048 collected 50 meters from MS#2 where the lower oyster unit was very distinctive in outcrop) suggest that the "older aminozone from the oyster unit" has been found "in place" (rather than being represented by reworked shells).

The spread of D/L values in the oyster unit could also be a consequence of groundwater flow and diagenetic alteration, which could also have affected shells in the overlying sand and serpulid units. Alteration fronts are visible in the measured sections, particularly at the boundary about 0.5 to 1.0 meter above the pink flag in MS#1, MS#2, and MS#3. The presence of shell ghosts throughout the upper part of the section (see MS#1 and MS#2 descriptions) indicates that there has been substantial loss of carbonate from the section. The contrast in porosity and lithology across the upper contact of the oyster unit results in perched ground water. Leaching and iron staining of shells in the oyster unit is common. High uranium content of both the shells and the sediment in the oyster unit has been noted (Mirecki and others, 1995; Kaufman and others manuscript; M. Lamothe, unpublished). Collectively, these features imply that some material was selectively released and translocated from the porous, upper sandy units to the tight underlying oyster unit. It is not clear how much of the alteration, leaching and translocation of material is related to the regional drawdown of the watertable in the last 30+ years (G. Johnson, pers. comm., 1995) and how much is a natural part of the subsurface geochemical system for the region. Nevertheless, in spite of the preliminary and speculative nature of these observations, it is important to emphasize that these processes, which clearly have affected the molluscan material in the section, have probably also affected the corals (and the U-series system) and the luminescence systematics (e.g., consistency of dose-rate). The spread of D/L values in the IIa, 95-043, and 95-044 samples also must be related to these diagenetic leaching effects.

## AAR Kinetic Modeling

Because AAR requires direct or indirect calibration for its use as a dating method, any interpretation of the D/L values in terms of time will depend on the accuracy of the calibration as well as the accuracy of the AAR results. The AAR data from Gomez Pit can be interpreted in both regional and local contexts. The regional context has been reviewed extensively (Wehmiller and Belknap, 1982; Wehmiller and others, 1988, 1992) and will not be addressed here. The local interpretation, in this case, will emphasize the <u>difference</u> between sample ages for the oyster and serpulid units, inferred from different kinetic models for racemization and different options for the ages of the calibration samples. This approach follows that given in Mirecki and others (1995: Table 4.1-2).

We use two kinetic models to provide model age estimates. Table 4.1-8 lists six D/L leucine values that span the range observed in the present study (with the exception of the Chowan River 95-038 samples). The different columns list age assignments for different choices of calibration ratios (either 80 ka or 125 ka calibrations are use) for two kinetic models: "B" for "Boutin non-linear model", presented in Wehmiller and others (1988); and "PB" for parabolic model, presented first by Mitterer and Kriausakul (1989). The Boutin model is applied directly to leucine D/L values; the parabolic model is applied to A/I values, converted from the appropriate leucine D/L value using the intrageneric conversion equations in Wehmiller and others (1988). Model runs B/1, B/2 and B/3 use a D/L leucine value of 0.25, 0.30, and 0.30,

D/L	B/1	B/1	B/2	B/2	B/3	B/3	PB1/	PB1/	PB2/	PB2/	PB3/	PB3/
Leu.	80	125	80	125	80	125	80	125	80	125	80	125
0.25 0.30 0.35 0.40 0.45 0.50	80 126 192 284 410 576	125 197 300 445 640 900	80 122 180 260 365	125 190 182 406 571	-52- 80 118 170 239	81 125 184 266 374	80 136 213 316 447 609	125 213 334 494 699 952	-47 80 125 186 262 357	73- 125 196 289 410 558	30- -51- 80 118 167 228	47 80 125 184 261 356

TABLE 4.1-8. KINETIC MODEL OPTIONS - LEUCINE<sup>1</sup>

<sup>1</sup>Ages in ky for different D/L leucine values, as predicted by different kinetic models: B = Boutin non-linear (see Wehmiller and others, 1988); PB = parabolic.

respectively, for the 80 and 125 ka calibrations. Model runs PB1, PB2, and PB3 use these same values for the two calibrations, respectively.

The results of the comparisons of the Boutin and parabolic kinetic models are shown in Figure 4.1-15. The difference in age estimates is much more dependent on the choice of calibration sample than on the choice of kinetic model. For example, if a D/L leucine value of 0.25 is used as either the 80 or 125 ka calibration, then a D/L leucine value of 0.40 would be interpreted to represent an age of either 300 ka or 475 ka, respectively, by either model (within ca.  $\pm$  5 percent, a range much smaller than the analytical range on an aminozone). Consequently, the following qualitative age estimates can be presented for the leucine aminozones observed in this study:

Using leucine D/L of 0.25 (the "best minimum value" for the serpulid unit) as an 80 ka calibration (the inferred age based on the U-Th dates of corals associated with the serpulid unit at 06076) predicts:

0.30 = ca. 125 ka 0.35 = ca. 200 ka (the mean value of 95-043 and 95-044?) 0.40 = ca. 300 ka 0.45 = ca. 425 ka ("best value" for IIc?) 0.50 = ca. 600 ka (95-048?)

If a leucine value of 0.35 (the mean of 95-043 and 95-044) is used as the 80 ka calibration, then it is possible to reach a leucine D/L of 0.50 by about 240 ka. Note that no corals from the sites of collection of 95-043 and 95-044 have been dated. Using the same D/L leucine value options with a 125 ka calibration will increase all of these age estimates by about 55-60 percent (a factor of 125/80).

Using conservative estimates of the "best" mean value for each of the Gomez Pit leucine aminozones, and assuming that there is as much as 15 percent variation (due to shell preservation effects), it seems appropriate to assume a mean leucine D/L of ca. 0.33 for 100 ka and to project the kinetics of Figure 4.1-15 to age estimates of ca. 220 ka (stage 7?) and ca. 300 ka



Figure 4.1-15. Kinetic model age estimates from Table 4.1-5. Models labelled "B" are for "Boutin nonlinear" (Wehmiller and others, 1988). Models labelled "PB" are for "parabolic kinetics" (see Mitterer and Kriausakuk, 1989). Three pairs of curves are shown for each model category - those with leucine values of 0.25, 0.30, and 0.35 "fixed" to calibration points of either 80 kyr or 125 kyr.

(stage 9?) for the leucine values of ca. 0.42 and 0.50, respectively. Although these age estimates for the older aminozones are slightly younger than those presented in Mirecki and others (1995), it must be emphasized that, at the present state of knowledge, the combination of analytical and kinetic model uncertainties for AAR methods, combined with unknown diagenetic variables, could easily result in any age estimate being "off" by 100 ka, even though the relative age differences for different aminozones might remain stable.

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## SUMMARY AND CONCLUSIONS

Results of this study add to our understanding of the relative reliability and comparability of the IRSL, U-series, and AAR geochronologic methods, and highlight specific problems. Comparability was enhanced because these methods were applied on the same measured sections, and because geologists and geochronologists worked side-by-side throughout the project, both in the field and in the lab. Results of this study also improve the geochronologic control on the Gomez Pit section of the U.S. Atlantic coastal plain sediments by significantly adding to the available age estimates on samples from this section.

The U-series and amino acid data obtained in this study duplicated most of the previous results. Although, some subtle differences in amino acid D/L values were encountered Some of the differences in AAR results can be ascribed to different sampling and analytical methods compared with the earlier work we have done at Gomez Pit (Mirecki, 1990), but some of these new results may be indicative of diagenetic effects that remain to be evaluated.

The twelve samples dated by the IRSL method are thought to have the same feldspar composition and their luminescence unstability would suggest that the ages reported should be considered as minimum ages, until a solution to anomalous fading can be found. The differences in ages between the different marine units is however significant.

Collectively the three dating methods indicate that there are at least two Pleistocene fossiliferous marine units preserved in the Gomez Pit section, one representing all or part of marine isotope stage 5, the other representing all or part of marine isotope stage 7 and/or perhaps stage 9. The apparent IRSL ages for the samples show three clusters: one at ca. 40-45 ka, one at 70-100 ka, and a single sample (VIR-1) yielded an older age of 160 ka. U-series results confirm earlier reports of ca. 70-80 ka ages on coral in fossiliferous units exposed at Gomez Pit and nearby pits. The AAR results indicate that the units above the oyster unit span from ca. 130 ka to 80 ka and younger, with the age of the serpulid unit at least 80 ka. Shells from the oyster unit(s) provide two age estimates that may correspond to deposition during marine-oxygen-isotope stages 7 and 9, but the effects of diagenetic alteration on these shells remains to be more fully evaluated.

Carbonate leaching and shell alteration are clearly evident in the section at Gomez Pit, despite the many "well-preserved" shells. The preservation quality of most of the specimens in the oyster unit indicates active water flow, staining, dissolution and possible precipitation, making this unit very complex both geochemically AND chronostratigraphically. The implications of these diagenetic processes for the accuracy of the U-series age estimates from Gomez Pit need to be fully evaluated, as it seems reasonable that the corals have not been "immune" from these processes.

The three most important things to getting a good age estimate are context, context and context, and these were supported by the results of this study. By having geologist and geochronologist present at the time of field reconnaissance and sampling, and in communication during lab analysis, the results have lower uncertainty by virtue of the fact that each participant witnessed the actions of the others.

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Appendix 🖋 🎵 Amino acid racemization data, 1995-1996 analyses , Gomez Pit collection

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Glu	0.177 0.197	0.159	0.215 0.214	0.196		0.278	0.170	0.181	0.220	0.222 0.245	0.181	0.163 0.150	0.203 0.210			0.191 0.238		0.240 0.240	0.257 0.220 0.227
Phe	0.359 0.365	0.262	0.493 0.267	0.269		0.409	0.220	0.270		0.237 0.355	0.254	0.197 0.203	0.282			0.342 0.378		0.396	0.329 0.328
Asp	0.632 0.627	0.499	0.608 0.598 0.587	0.601 0.610		0.625 0.640	0.532	0.575	0.614	0.539 0.593	0.537	0.540 0.547	0.602 0.583		0.572	0.579		0.559 0.586	0.574 0.622 0.582 0.563
Leu	0.404 0.408	0.235 0.245	0.371 0.368 0.362	0.364 0.368		0.397 0.393	0.283	0.288	0.380 0.392	0.372 0.369	0.296 0.296	0.246 0.261	0.351 0.346		0.244	0.354 0.364		0.407 0.398	0.360 0.376 0.306 0.306
A/I gc	0.390 0.369	0.207	0.369 0.344 0.361	0.378 0.354		0.348 0.385		0.215	0.358	0.283 0.303	0.238 0.235	0.216 0.225	0.317 0.330		0.216	0.328 0.347		0.381 0.346	0.301 0.287 0.275
Val	0.20 <b>4</b> 0.200	0.130	0.171 0.172 0.172	0.177 0.173		0.201 0.192		0.126	0.150 0.215	0.234 0.208	0.161 0.161	0.132 0.143	0.197 0.206		0.142	0.226 0.200		0.197 0.209	0.207 0.154 0.139
Ala	0.483 0.497	0.439	0.420 0.411	0.436 0.431		0.483 0.501	0.390	0.420	0.450 0.495	0.606	0.462 0.457	0.430	0.513 0.456	104 0	0.453	0.521 0.485		0.563 0.556	0.516 0.431 0.432
comments		split for hplc and gc	split for hplc and gc gram modifications	gram modifications gram modifications	awlful gc run	CAUTION CAUTION	small hplc/gc	railed no inji	small hplc/gc	small hplc/gc	small hplc/gc too big for asp-glu		small peaks		n with aux he onli!!			CAUTION CAUTION	split for hplc and gc split for hplc and gc CAUTION CAUTION
A/I hpic	0.335 0.335	0.150	0.290 s 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.290 0.2	0.290 0.290 0	0.130	0.295 0.295	0.155	0.155	0.340 0.340	0.280 0.280	0.230	0.190 0.190	0.270 0.270		0.201	0.285 0.285		0.340 0.340	0.280 0.280 0.235 0.235
Sample	jw95-043-1a jw95-043-1a	jw95-043-1b outer c jw95-043-1b outer c	jw95-043-1b middle jw95-043-1b middle jw95-043-1b middle	jw95-043-1b middle jw95-043-1b middle	jw95-043-1b outer	jw95-043-1c jw95-043-1c	jw95-043-1c-x	Jw95-043-16-X Jw95-043-1c-X	jw95-043-1c-y inner jw95-043-1c-y inner	jw95-043-1c-y middle jw95-043-1c-y middle	jw95-043-1c-z jw95-043-1c-z	jw95-043-8-1-x jw95-043-8-1-x	jw95-043-8-1-y jw95-043-8-1-y		jw95-043-8-2-x	jw95-043-8-2-y jw95-043-8-2-y		jw95-044-4 jw95-044-4	jw95-044-3 jw95-044-3 jw95-044-6 jw95-044-6
Run	22	86 91	85 99 1	2 0	104	67 3	96	5 4	97 7	101 15	98 23	18 21	16 46	76	88	39 44		60 62	14 15 66 71
Lab No	960143 960143	960062 960062	960063 960063 960063	960063 960063	960064	950511 950511	960086	980096	960087 960087	960088 960088	960089 960089	960144 960144	960145 960145	060146	960146	960147 960147		950502 950502	950528 950528 950509 950509
Date	3/5/96 3/5/96	2/15/96 2/16/96	2/15/96 2/21/96 2/26/96	2/26/96 2/26/96	2/22/96	12/7/95 12/12/95	2/20/96	2/29/96	2/20/96 2/28/96	2/21/96 3/1/96	2/20/96 3/6/96	3/1/96 3/5/96	3/1/96 3/12/96	3/0/0/5	3/9/96	3/9/96 3/11/96		12/4/95 12/4/95	12/19/95 12/19/95 12/7/95 12/11/95
	- N N 4	8 4 6 2	o 0 ⊑ 2	5 4 5	9 5 5 6 5 6 5 6 5 6 5 6 6 5 6 6 6 6 6 7 8 6 6 7 8 6 6 7 8 6 6 7 8 6 7 8 7 8	2 2 2 2	2 7 2	0.81	8 8 8	3 5 8 8	38 35 34	39 38	<del>6</del> <del>6</del> 6	4 4 7 7 4 7 7 4 7	42	46 47	48 49 50	51 52	53 55 57

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Gomez 1995 Samples

Gomez 1995 Samples

Głu	0.227 0.216 0.218 0.229	0.290 0.321 0.422 0.380 0.345	0.345 0.305 0.303 0.324 0.324 0.327 0.327 0.327	0.815 0.860 0.857 0.857 0.857 0.699 0.793	0.333 0.357 0.346 0.337 0.333	0.237 0.233	0.246 0.246 0.239
Phe	0.356 0.326 0.315 0.334	0.512 0.548 0.554 0.554	0.395 0.411 0.507 0.439 0.441 0.458	0.849 0.891 0.858 0.866 0.866 0.866 0.814	0.468 0.538 0.514 0.459 0.449	0.315 0.330	0.396 0.368 0.490
Asp	0.600 0.591 0.595 0.602	0.654 0.696 0.733 0.690 0.690 0.743	0.654 0.672 0.670 0.672 0.645 0.662 0.662 0.652	0.907 0.919 0.906 0.906 0.914 0.912 0.910	0.690 0.687 0.700 0.691 0.691	0.541 0.542 0.619 0.604	0.616 0.608
Leu	0.345 0.353 0.346 0.348	0.455 0.445 0.446 0.463 0.472 0.483 0.477	0.454 0.463 0.445 0.434 0.433 0.439 0.434 0.432 0.432	0.857 0.885 0.914 0.898 0.896 0.899 0.869 0.869 0.879	0.519 0.509 0.514 0.502 0.511	0.339 0.344 0.332 0.342	0.428 0.426 0.365
A/I gc	0.306 0.308 0.343 0.316	0.496 0.496 0.503 0.484 0.484 0.473 0.483	0.530 0.320 0.436 0.419 0.421	1.260 1.143 1.260 1.140 1.022	0.473 0.452 0.444 0.463 0.452	0.267 0.262 0.243	0.405 0.415 0.321
Val	0.202 0.208 0.197 0.205	0.302 0.303 0.325 0.325 0.320 0.311 0.319	0.255 0.251 0.333 0.284 0.284	0.786 0.698 0.832 0.836 0.859 0.841 0.800 0.827	0.327 0.376 0.364 0.358 0.358	0.191 0.176 0.191 0.189	0.218 0.206 0.188
Ala	0.444 0.386 0.440 0.454	0.661 0.618 0.638 0.638 0.612 0.613 0.613 0.621	0.692 0.643 0.631 0.724 0.646 0.678	0.887 0.890 0.991 0.9956 0.955 0.955 0.895 0.895	0.685 0.741 0.695	0.659 0.619 0.767	0.610 0.623
comments	0 BASELINE DROPPE	<ol> <li>5 imez nrc oyster bed</li> <li>5 imez nrc oyster bed</li> <li>85 imez nrc oyster bed</li> <li>80 imez nrc oyster bed</li> <li>90 imez nrc oyster bed</li> <li>90 imez nrc oyster bed</li> <li>90 imez nrc oyster bed</li> </ol>	0 imez nrc oyster bed 0 imez nrc oyster bed 00 imez nrc oyster bed	<ul> <li>0 gomez nrc Chowan</li> <li>00 gomez nrc Chowan</li> </ul>	50 NOISY BASELINE 50 50	<ul> <li>70 pilt for hplc suspect</li> </ul>	40 40 00 (s big some overload
A/I hplc	0.24 0.24 0.24 0.24	0.43 0.43 0.43 0.43 0.43 0.43 0.43	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	1.30 1.30 1.30 1.30 1.30 1.30 1.30 1.30	0.0 94.0 94.0 94.0 94.0	0.27 0.27 0.27 0.27	0.34
Sample	jw95-044-6 jw95-044-6 jw95-044-6 jw95-044-6	Jw95-035-1 Jw95-035-1 Jw95-035-1 Jw95-035-2 Jw95-035-2 Jw95-035-2	Jw95-037-1 Jw95-037-1 Jw95-037-2 Jw95-037-2 Jw95-037-2 Jw95-037-2 Jw95-037-2	jw95-038-1 jw95-038-1 jw95-038-1 jw95-038-2 jw95-038-2 jw95-038-2 jw95-038-2	jw95-046-1a articulated jw95-046-1a articulated jw95-046-1a articulated jw95-046-1a articulated jw95-046-1a articulated	jw95-046-13 jw95-046-13 jw95-046-13 jw95-046-13	Jw95-046-13a jw95-046-13a jw95-046-13b
Run	66 67 74 78	19 28 28 25 31 31	20 21 21 21 22 23 50 52 52	32 35 36 37 37 38 37 37 37 38 37 37 37 37 37 37 37 37 37 37 37 37 37	65 68 76 81 81	61 72 2	43 48 28
Lab No	960055 960055 960055 960055	950439 950439 950439 950440 950440 950440 950440	950441 950441 950441 950442 950442 950442 950442 950442 950442	950443a 950443b 950443b 950443b 950444b 950444b 950444a 950444a 950444a 950444a 950444a	960057 960057 960057 960057 960057	950503 950503 950503 950503	960148 960148 960149
Date	1/28/96 1/29/96 1/31/96 2/1/96	9/15/95 9/19/95 9/18/95 9/19/95 9/19/95 10/3/95	9/15/95 9/18/95 9/20/95 10/12/95 10/12/95 11/28/95 11/29/95 11/29/95	10/4/95 10/5/95 10/9/95 10/9/95 10/9/95 9/15/95 9/18/95 9/18/95	1/28/96 1/30/96 2/1/96 2/13/96 2/13/96	12/4/95 12/6/95 12/11/95 12/12/95	3/11/96 3/12/96 3/7/96
	58 60 63 79 63 79 78 78 78 78 78 78 78 78 78 78 78 78 78	65 66 67 7 7 68 7 7 7 68 7 7 7 68 7 7 7	88 88 83 8 9 9 9 9 7 7 7 7 7 7 7 7 7 8 8 8 8 8 8	88 89 99 89 99 99 99 99 99 99 99 99 99 9	99 101 102 103 105 105	106 108 1109	112 113 113

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						Gomez	1995 Sample	<i>i</i> 0					Tue, Al	9
	Date	Lab No	Run	Sample	A/I hplc	comments	Ala	Val	A/I gc	Leu	Asp	Phe	Glu	
115 116 117	3/12/96	960149	45	jw95-046-13b	0.300		0.519	0.187	0.348	0.363	0.573			
118	1/30/96	960056	70	jw95-046-17	0.370	smail peaks				0.429	0.602			
119	1/30/96	960056	71	jw95-046-17	0.370		0.783	0.282	0.338	0.437	0.628	0.468	0.322	
120 121 122	1/31/96	960056	75	jw95-046-17	0.370		0.635	0.286		0.447	0.653	0.544	0.309	
124	12/7/95	950512	68	iw95-046-21	0.322 pil	t for hold suspect				0.415	0.589			
125	12/8/95	950512	69	iw95-046-21	0.322 pil	t for hold suspect		0.236		0.400	0.636	0.445	0.306	
126	12/8/95	950512	70	jw95-046-21	0.322 pil	t for hplc suspect		0.227		0.390	0.676	0.410	0.337	
127 128														
129	3/6/6	960150	40	jw95-046-21a inner	0.490 <i>i</i> rl	oad of tater peaks	0.611	0.289	0.495	0.481				
130	3/11/96	960150	41	jw95-046-21a inner	0.490	-	0.546	0.303	0.535	0.484	0.665	0.398	0.285	
131	36/1/8	960151	29	jw95-046-21a middle	0.450		0.679	0.298	0.473	0.483	0.639			
132	3/9/9	960152	25	jw95-046-21b	0.440	small				0.450	0.625			
133	3/11/96	960152	42	jw95-046-21b	0.440		0.605	0.282	0.441	0.445			0.288	
134 135														
136	12/1/95	950504	59	iw95-048-7	0.440 nil	t for hole susnect	0 768	0315	0.551	0.492	0.633			
137	12/6/95	950504	63	w95-048-7	0.440 pil	t for hpic suspect	0.730	0.320	0.467	0.486	}	0.504	0.321	
138														
139	12/14/95	950527	2	jw95-048-4a	0.460 sl	olit for hpic and gc					0.693	0.477	0.339	
140	12/14/95	950527	9	jw95-048-4a	0.460 si	blit for hplc and gc	0.726	0.321	0.433	0.544				
141	1/25/96	950529	57	jw95-048 4b	0.480 le	mix with 960057	0.681	0.328	0.563	0.568	0.742			
142	1/25/96	950529	58	jw95-048 4b	0.480 le	mix with 960057	0.638	0.321	0.513	0.552		0.529	0.348	
143	1/26/96	950529	63	jw95-048 4b	0.480 le	mix with 960057	0.728	0.332	0.537	0.537	0.702	0.541	0.363	
144	1/26/96	950529	64	jw95-048 4b	0.480 le	mix with 960057	0.720	0.313	0.540	0.547	0.690	0.495	0.315	
145														
146		960058		jw95-048 5a	0.525	no gc data								
147		950526		jw95-048 6	0.470	no gc data								

no gc data no gc data

0.525 0.470

jw95-048 5a jw95-048 6

960058 950526

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						Gomez & N	Moyock pre	95					Tue, A
	Date	Lab no	Run no	Sample	A/I hplc	Comments	Ala	Val	A/I gc	Leu	Asp	Phe	Glu
+0040078001	2/28/96 2/19/96 2/19/96 2/19/96 2/21/96 2/21/96 2/19/96 2/19/96	960076 960077 960077 960077 960079 960079 960079 960079	8 92 100 94 95	gp278 x small pks gp278 y inner gp278 y inner gp278 y innel gp278 y middle gp278 y middle gp278 z	0.165 0.165 0.165 0.165 0.225 0.225 0.225	lit for hplc and gc lit for hplc and gc lit for hplc and gc nice run small hplc/gc nice run nice run lit for hplc and gc lit for hplc and gc	0.393 0.438 0.490 0.472 0.472 0.472 0.496 0.507 0.485	0.121 0.124 0.175 0.175 0.155 0.155	0.193 0.179 0.296 0.293 0.175 0.234 0.252	0.203 0.206 0.302 0.306 0.202 0.198 0.283 0.283	0.512 0.538 0.536 0.476 0.504 0.504	0.207 0.240 0.291 0.249	0.159 0.160 0.189 0.162
5 t t t t t t t	1993 1993	930172 930173		jw89-104 jw89-106	0.190	lone with sp, mck lone with sp, mck		<b>.</b>					
22 23 23 23 24 28 25 25 25 25 25 25 25 25 25 25 25 25 25	2/15/96 2/16/96 2/15/96 2/16/96	960080 960080 960081 960081 890027 890028	888 888 89	jw89-121-b inner jw89-121-b inner jw89-121-b outer jw89-121A JW89-121A JW89-121B	0.240 0.240 0.200 0.200 0.160 0.180	lit for hplc and gc lit for hplc and gc small lit for hplc and gc	0.348 0.350 0.443	0.148 0.133 0.142	0.262 0.262 0.217	0.294 0.291 0.268 0.256	0.595 0.563 0.494	0.269 0.241 0.357	0.172 0.185 0.166 0.166
22 33 33 33 33 33 33 33 33 33 33 33 33 3	7/19/95 7/19/95 7/19/95	950337 950337 950337 950338 950338 950334 950335 950335	N & O	Jw89-139-a1 Jw89-139-a1 Jw89-139-a1 Jw89-139-a2 Jw89-133A 1 Jw89-133A 1 Jw89-133A 2	0.203 0.203 0.160 0.160 0.160 0.130 0.135 0.235	M ONeal Gomez M ONeal Gomez M ONeal Gomez M ONeal Gomez M ONeal Gomez M ONeal Gomez M ONeal Gomez	0.434 0.438	0.144 0.156	0.234	0.252 0.251	0.534	0.233 0.240	0.198 0.190
335 36 33 33 33 33 33 33 33 4 4 4 4 3 3 3 3		950339 950340 960070 950341 950342		jw89-141a-1 jw89-141a-2 jw89-141a-3 jw89-148A-1 jw89-148A-2	0.200 0.160 0.175 0.175 0.230 0.190	M ONeal Gomez M ONeal Gomez pollard M ONeal Gomez M ONeal Gomez							
5 1 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	7/20/95 7/20/95 7/20/95	950393 950393 950393 950394 950394 890005	9 <u>∓</u> 8	jw88-46-1-1 jw88-46-1-1 jw88-46-1-1 jw88-46-1-2 jw88-46-1	0.369 0.369 0.380	M ONeal Gomez M ONeal Gomez M ONeal Gomez M ONeal Gomez GTF 1989	0.738 0.831 0.688	0.286 0.309 0.287	0.439 0.399 0.437	0.419 0.407 0.410	0.634 0.650	0.363 0.423 0.384	0.293 0.262 0.262
53 55 57	3/1/96	960142 890040 890039	17	jw89-163 jw89-163 jw89-162	0.310	LARGE PEAKS 9 gtf opa effects 99 gtf opa effects	0.632	0.306	0.479	0.461			

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Gomez & Moyock pre '95

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Б	291	289	289	320			318			281	040	280					•	241	695	191	load	053	123	861	341	613	422	l				
Ū	Ö	o.	Ö	Ö			o.			C	o c	i c	5					0	0.2	[ 2		0.2	0.1	E	16	0	0 0	5				
Phe		0.416	0.505	0.453			0.439			0.468	0.385	0.417	5				•	0.289	0.272	0.294	oload	0.2498	0.174	[.2064]	oload	0.2412	0 2908	000-00				
Asp	0.634	0.621	0.632	0.629	0.652	0.616	0.618			0.637	0.619	0.622					[.72]	0.549	0.548	0.5507	oload	0.5422	0.5356	0.5526	oload	0.4917	pload					
Leu	0.407	0.413	0.416	0.413	0.417	0.417	0.408			0.404	0.401	0.414					0.317	0.323	0.314	0.318	0.309	0.309	0.224	0.311	0.312	0.220	0.265					
A/I gc	0.404		0.440	0.377	0,414	0.424	0.408			0.374	0.381	0.368					0.305	0.300	0.305	0.296	0.302	0.316	0.202	0.274	0.244	0.185	0.214					
Val	0.269		0.241	0.271	0.277	0.265	0.282			0300	0.287	0.282					0.187	0.191	0.164	0.192	0.186	0.166	0.117	0.149	0.159		0.134					
Ala	0.609		0.589	0.614	0.602	0.622				0690	0.691	0.702					0.494	0.518	0.582	0.485	0.496	0.480	0.297	0.446	0.427	0.373	0.502					
Comments	olit for hpic and gc	olit for hpic and gc	olit for hpic and gc										12/95 run				ko split - moyock	:ko split - moyock	ko split - moyock	olit for hplc and gc	olit for hpic and gc	olit for hplc and gc	olit for holc and gc	larde beaks								
A/I hplc	0.385	0.385	0.385	0.370	0.370	0.370	0.370			0.370	0.370	0.370	0.360				0.260	0.260	0.260	0.260	0.260	0.260	0.160	0.235	0.235				0.191	0.251	0.229	0.223
Sample	jw89-170	jw89-170	jw89-170	jw89-170	jw89-170	jw89-170	jw89-170			JW89-173	JW89-173	JW89-173	JW89-173				JW93-61-4	JW93-61-4	JW93-61-4	JW93-61-4	JW93-61-4	JW93-61-4	jw93-61-4x	jw93-61-4y inner	jw93-61-4y inner	w93-61-4y middle	iw93-61-4z		jw93-61-1	jw93-61-2	jw93-61-3	jw93-61-4
Run no	4	13	23	59	60	61	62			72	73	84					19	21	25	26	27	28	თ	10	47	1	12					
Lab no	950515	950515	950515	960060	960060	960060	960060			960061	960061	960061	950416				950234	950234	950234	950234	950234	950234	960082	960083	960083	960084	960085		930177	930178	930179	930180
Date	12/14/95	12/18/95	12/22/95	1/25/96	1/25/96	1/26/96	1/26/96			1/31/96	1/31/96	2/14/96					5/4/95	5/5/95	5/9/95	5/9/95	5/10/95	5/10/95	2/28/96	2/28/96	3/12/96	2/29/96	2/29/96		1993	1993	1993	1993
	59 59 60	61	62	63	64	65	66	67	00	22	71	72	73	74	75	9	1	78	79	80	81	82	83	84	85	86	87	88	89	06	91	92

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Appendix III Gomez Pit survey data, Sept. 1995

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Gomez Pit Survey 9/95	
Short Exposure	
Bottom of Section (refd to H	13 an map - 2 pts an convers it outcrop)
D(m) Elev (m MSL)	
70.1 - 1.16	
76:6 -1:76	
Top of Oyster Bed	TL Sites
D Eler	D <u>Elev</u>
45,5 Oisl	45.7 -1.03 in plane of outerap
53,2 0,46	39.5 1.49
59,8 0,54	40,0 2.38 in cross plane 1
63.1 0.46	42,2 9,80 -
Pink Section	
D Ebu	
54.2 1.79	
45,2 1.69	
41,3 1.56	
39.8 1.69	
Blue Section	
D Elev	
42.2 5.13	
48.1 5.36	
55,2 5,34	
62.7 5.32	
Top of Outcrop	
D Eles	
54.4 8.25	
48,4 8,19	
44,6 7,86	

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Gomez Pit Survey 9/95

Long Exposure - Top of Section

D(m)	Eles LM MSL)	Blue S	ection	Orange Section
8.1	7.79	D	Eles	D Eles
29.3	7.04	153,3	5.08	163.6 4.37
57,3	7.41	145.1	4.96	
83.4	7.72	140.1	5.03	100.7 4.22
90,8	7.75	135.5	4,91	
111.3	7.84	131.3	5,08	158.1 4.61
126.1	7,83	125.9	5,33	155.9 9.76
145.2	7.77	122.6	5.33	/53.0 5.58
161.9	7.32	118.5	5.32	/52.5 5.87
166:5	5.95	114.2	5.55	151.9 6.15
		108.9	5,40	Top of Oxster Bod
Pink Sc	action_	105.6	5,39	D Bles
D	Elev	90,7	5.31	153.6 -0.96
<u> </u>	191	87.3	5,34	145.2 -0.88
12,1	1.77	79.7	5,53	135.6 -0.92
70.0	1,73	61.1	5.23	115,8 -1,03
4016	1.23	56.3	5.49	103.3 -0.97
73,3 50 0	1.69	46.3	5.20	95,8 -0,93
50.0		41.9	5.27	89.9 -0.95
1212	1.30	37, 7	5.24	78.2 -0.96
100	1.42	32.9	5.52	44.0 -0.93
עיניס קמנו	1.37	26.6	5.42	53,3 -1,03 Z JEW yel
10,7	1,53	12.5	5,14	41.6 -1.00 S flags
010,4 A A A	1.57			25.8 -0,94 - corner
gan	1.33	Green Sec	tion	Retta R D'+
1030	143	DE	ler	D Ela (
105.0		161.7 4	1,16	$\frac{1}{100} = 258$
110.3	1 39	160.6 4	1.14	5.7 - 7.4
116.9	152	158.6	1.06	973 - 747
1239	1.46	157.9 4	1.09	13.2 - 2.73
129.1	1.57	156.1 4	1.09	154.9 -1.54
174.4	1.104	152.3	3.67	1584 -1.29
1422	1 61	149.5	3.93	
147. J	1.41			
153.9	193			

159.7 1.86





Appendix IV Thermoluminescence data, 1989 sampling, Gomez Pit



Fax #302-831-4158	# pages incluant calle-ci
Tvléphone (514) 987-8628	Fax (514) 987-7865 E-Mail lamothe.michel@ugam.td
Lux	Laboratoire de Luminescence de l'Université du Québec à Montréal Département des Sciences de la Terre - GEOTERAP C.P. 8688, Succ. Centre-Ville, Montréal, Québec, Canada, H3C 3P8

Dr John Wehmiller Dept. Geology University of Delaware

Dear John,

Thank you for your last letter and reprints. It was a good thing that you renewed contact recently since it gave me the opportunity to look at the Virginia data again. As I mentioned to you last year, the preliminary ages we obtained were a bit younger that expected. This might be due to downward percolation of fines from the top since we analyzed the so-called 4-8 µm fine grains". I am annexing here the preliminary Infrared Stimulated Luminescence integrated growth curves. The numbers are based on several approximations, such as the alpha efficiency, the cosmic dose (~ 15 mGy/ka), and the water content ( ~ 25%). For VIR-2, the reproducibility was not great and also this sample has much higher U and Th abundances than the other two. Could you tell me why this is? I think that the best thing to do now is to isolate the feldspar and qaurtz sand sized grains, which we do here in routine, and measure ages on them in order to confirm or refine the data. I have realized that in our business, we have to measure ages on several types of minerals of different grain sizes in order to get the best age estimate. I will send you some of my reprints in which you will see how, I believe, we could date samples in which are evidence of "intrusion" of older or younger grains.

I have only a "small" problem: I do not have any money. Do you think you could participate financially in the project, for an amount of ~ CDN\$ 500-1000? I could then pay one of my student assistant who would do the mineral separation and the mounting and irradiation of the sample. I am sure we could have something really solid for next spring. This project is actually very interesting in the OSL point of view since there is quite a dispute about the underestimation of ages from older interglacial sediments, this being particularly raging in Europe. Your project is a great supply of material to test the technique.

Best regards.

Michel Lamothe Professeur

