THE 4300-YR ¹⁴C AGE OF GLYPTODONTS AT LUJÁN RIVER (MERCEDES, BUENOS AIRES, ARGENTINA) AND COMMENTS ON 'DID THE MEGAFAUNA RANGE TO 4300 BP IN SOUTH AMERICA' BY CIONE ET AL.

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INTRODUCTION

The glyptodont (Glyptodontidae Burmeister 1879, in Ameghino 1889), a giant cousin of the *arma-dillo*, has long been thought to have disappeared in South America at least 10,000 years ago at the end of the Pleistocene. There are indications that the glyptodont had some interaction with ancient hunter-gatherers peoples (Politis et al. 1987; Politis 1995), but the precise time of its extinction has never been well established. Most recently, a brief mention of ¹⁴C dates of 7500 to 6500 BP (Geotimes 1996) for glyptodont remains discovered at La Moderna (Azul Department, Buenos Aires province, Argentina), has aroused excitement because the new younger dates have changed the traditional idea about the survival and extinction of this beast.

Rossello et al. (1999) have report an even younger ¹⁴C age of 4300 yr for the glyptodont bone fossils found in the type locality of the famous Luján Formation (Ameghino 1914; Bonaparte 1958), the top stratigraphic levels of the Pampean loess deposits (Sayago 1995; Iriondo 1997). The dated fossil remains consist of dermal plates of lateral carapace and caudal vertebrae of *Glyptodont clavipes* Owen 1838 of the family Glyptodontidae Burmeister 1879 (for more details, see Ameghino 1889; Cione and Tonni 1995, etc.). In association with these fossil remains were also found bones of tarsus, ribs, fragments of pelvic girdle and osteoderms, probably corresponding to a specimen of *Mylodontidae* (Ameghino 1889). In South America these families have a wide biological distribution from the Lower Miocene to the Pleistocene (Cione and Tonni 1995; Prado and Cerdeño 1998).

The new 4300 yr age (Rossello et al. 1999) certainly provides some important implications regarding the determination of the stratigraphical boundary between the Pleistocene and Holocene, as well as the life patterns of ancient peoples of the Argentinean Pampas in South America. The studied glyptodont bone fossils of one specimen were discovered at Mercedes City in the Buenos Aires province, Argentina. The excavation site is located at 59°20'W and 34°40'S, and at 38 m above sealevel from the southern bank of the Luján River, about 200 m upstream from the discharge of the pluvial channel at Mercedes City. They were found in living position (upright) within the Luján Formation and partially eroded and covered by a thin bed of fluvial conglomerates made up of small carbonate rolling pebbles and a recent 1.5 m thick level of massive flood sediments with a welldeveloped soil profile. In equivalent and closer levels, is very frequent finding other remains of the same or different specimens.

Since Ameghino (1889) first introduced a stratigraphic scheme, many others have been proposed which resulted in a proliferation of local stratigraphies, often with confusing regional correlation. In the last 25 years, several authors have carried out stratigraphic studies and continued to revise the already complex stratigraphic and paleontological nomenclature of the Pampean region (Fidalgo et

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al. 1975; Marshall et al. 1984; Zárate and Fasano 1989; Clapperton 1993; Cione and Tonni 1995, etc.). New names as well as older ones have often been applied inappropriately to the same units. Stratigraphic correlations based on lithological characteristics have been very difficult because variable depositional environments might have resulted in different types of sediments deposited in the same period.

The Luján Formation is a lithoestratigraphical unit defined by Fidalgo et al. (1975) taken the old Ameghino's name from the Luján River. It is a few meters thick, composed of pale green or yellowish massive to locally slightly stratified clayey siltstone sediments (Ameghino 1889). These sediments were reworked from the underlying loessic Pampa Formation and redeposited in shallow water ponds or fluvial valleys. It is overlay by the Platense Formation that shows radiocarbon age of 2900 ± 40 yr obtained on gastropod shells (Fidalgo et al. 1975).

Despite some paleomagnetic studies (e.g. Orgeira et al. 1998) and a few absolute age measurements (Fidalgo et al. 1975; Gómez et al. 1987; Cione and Tonni 1995; Figini et al. 1995), the ages of the loessic Pampean sequences (e.g. Gallet et al. 1998) have been estimated mainly based on fossil vertebrate assemblages (Pascual 1984). However, the large biochronological span of the fossils usually makes accurate chronostratigraphic assignment difficult. Pascual et al. (1966) defined a variety of land-mammal ages for the Cenozoic era based on the faunal sequence originally proposed by Ameghino (1914). In this sense, three main criteria were taken into consideration to define these ages: 1) the stage of evolution of the taxa, 2) the change of faunal associations through time, and 3) the first or last appearance of the taxa in fossil record (Pascual, 1984). The Lujanian, whose name is derived from the Luján River at this locality, is the youngest land-mammal age. It is characterized by the last record of a large number of taxa (i.e. Proboscidea, Notoungulata, Glyptodontidea, Megatherioidea, *Litopterna*, Equidae, *Smilodon*) with the appearance of many new forms which persist to the present (Marshall et al. 1984; Tonni et al. 1985; Zárate and Fasano 1989). Collantes et al. (1993) also include Mylodontinae, Mastodon, Camelidae and Rodentia in this faunal association. The paleontological criterion has been almost the only one used so far to define both the Pliocene-Pleistocene and the Pleistocene-Holocene boundaries. The latter is marked by the extinction of almost alllarge mammalian herbivores and their specialized predators such as ground slots, glyptodonts, proboscidean horses, notoungulates, litopterns, and sabertooth cats (Marshall et al. 1984).

The studied remains were unearthed by JL Petrocelli (kindly helped by the *Amigos del Río Luján* group) and immediately stored to avoid any potential problem of contamination by radiation. Two months after, the ¹⁴C age measurements were conducted in the Radiocarbon Laboratory of Department of Geology, National Taiwan University (Taipei, Taiwan).

The analytical procedures were as follows.

1. *Pretreatment-collagen extraction* (following the procedures of Longin 1971): a) the bone sample was washed with deionized water, then dried; b) the sample was crushed and pulverized to particle size $\leq 2 \text{ mm}$; c) repeatedly added 1 *N* HCl to dissolve the inorganic fraction; decant the solution and collected the colloidal solution which contains insoluble collagen; d) stood still to settle down the collagen fraction and then centrifuge to concentrate collagen, e) decant the upper clear solution and retain the gelatinous collagen fraction; f) repeatedly washed the collagen fraction with 0.5 *N* NaOH and deionized water till the color of the collagen fraction became white; g) acidified the collagen with 1 *N* HCl to pH ≈ 4, then repeatedly washed it with deionized water several times until pH ≈ 6.5.

- Benzene synthesis: a) The collagen sample was dried and combusted in oven and 0.979 gm of benzene sample was obtained. b) Oxalic acid standard reference material used is NBS SRM 4990C, Oxalic acid (HOxII).
- 3. *Counting Activity*: measured using two Quantulus[®] 1220 scintillation counters. a) NBS HOXII = 52.898 cpm (for 3.9446 gm benzene), background = 0.553 cpm, bone sample = 6.261 cpm (for 0.979 gm bone benzene, which was diluted by dead benzene to 3.9445 gm), b) Counting times: counting time of HOXII standard = 1481 min, counting time of background = 2961 min, counting time of bone sample = 2221 min.
- 4. Age: 4370 ± 90 BP (first counting), 4250 ± 80 BP (second counting), conventional age = 4310 ± 90 BP (average).
- 5. $\delta^{13}C$ (bone) = $-18.3 \pm 0.1 \%_0$, $\delta^{13}C$ (HOxII) = $-17.74 \pm 0.1\%_0$.

The key issue is that Cione et al. (2000) questioned the validity of our measured age of 4300 yr and suspected that laboratory contamination by young carbon might be the cause for the young age. We shall argue that this is not likely to happen in our well-controlled laboratory. Indeed, there are often doubts or problems about ¹⁴C dates of collagen from poorly preserved bone samples. In order to minimize possible contamination, the bone sample was repeatedly washed and generously decanted. Now, if Cione et al.'s idea is correct and the true age is assumed to be 11,400 yr (about two half-lives of ¹⁴C), then a simple calculation would show that, in order to lower the age from 11,400 to 4300 yr, our sample must have been contaminated by about 45% of "young" or modern carbon (see Appendix for demonstration). If the contamination is not "modern" carbon, then the percentage will be even higher. Such a high level of contamination is absolutely impossible to occur during the analysis.

Cione et al. mentioned an example of a 100 ka sample that is somewhat misleading concerning the percentage of contamination. Of course, a sample of actual age of 100 ka may yield an apparent ¹⁴C age of 37,000 yr if it is contaminated by 1% of "modern" carbon. This can be easily shown by a simple calculation. In any case, 1% contamination for a very old object does not have the same effectiveness of 1% contamination for a young object.

The second point is more geological. Rossello et al. (1999) dated the original Luján Formation at the key locality defined by Ameghino (1889, 1914). However, the Cione et al.'s ages are referred to completely different outcrops about 200 km to the southeast. In their Figure 2 there is no indication about the stratigraphic position of the glyptodont. Besides, a glyptodont found in one place does not necessarily mean that the stratum has the same age as that of the second place where another glyptodont is found. Furthermore, they mentioned many ¹⁴C dates, are they also well controlled and of good quality? Their ages for the Holocene Las Escobas Fm range from 7890 ± 343 to 3050 ± 160 yr. They only said that the younger dates correspond to upper levels and the older to lower levels. But is it true that the positions of the animals correspond well with their ¹⁴C dates? We also like to emphasize that a few years ago, ¹⁴C dates of 7500–6500 yr. were reported for glyptodont remains discovered at La Moderna (Azul Department), west of Buenos Aires (Geotimes 1996). There is no reason to believe that the glyptodont must be older than 10,000 yr.

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APPENDIX

Assuming the true age is 2 half-lives = 11,400, and we put in *x* Co as contaminant. Using t = $1/\lambda \times \ln(\text{Co/C})$ (we may use the activity equation, also):

For the non-contaminated sample

$$2 \times \text{half-lives} = 1/\lambda \times \ln(\text{Co/C}). \tag{1}$$

For the contaminated sample

$$4300 = 1/\lambda \times \ln(\text{Co/C'}) = 1/\lambda \times \ln(\text{Co}/(1-x)\text{C} + x \text{ Co}).$$
(2)

From Eq. (1)	Exp $(\lambda \times 2 \text{ half-lives}) = \text{Co/C}$.	(3)

From Eq. (2)
$$\operatorname{Exp} (\lambda \times 4300) = \operatorname{Co} / ((1-x) \operatorname{C} + x \operatorname{Co}).$$
 (4)

Eq. (3) / Eq. (4)

$$Exp (\lambda \times 6840) = ((1-x) C + x Co) / C = (1-x) + x (Co/C) = (1-x) + 4x$$
(5)

because Co/C = 4 for the true age of 2 half-lives. Therefore, 2342 = 1 + 3x, thus, x = 0.447 = 45% ($\lambda = 0.00012444 \text{ yr}^{-1}$).