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# Growth process in an elephant tusk: Age estimations based on temporal variations in bomb-radiocarbon content



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BEAM INTERACTIONS WITH MATERIALS AND ATOMS

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#### ABSTRACT

In this study, <sup>14</sup>C analysis by accelerator mass spectrometry (AMS) was applied to age estimation based on temporal variations in bomb-produced-<sup>14</sup>C contents of a full elephant tusk registered at Kyushu University. The tusk measured 175 cm long and 13.8 cm in diameter at the root. Thirty tusk-fragment samples were used for <sup>14</sup>C analysis with AMS to estimate the formation ages of different positions according to catalogued global <sup>14</sup>C contents (F<sup>14</sup>C). The F<sup>14</sup>C value of the tip of the tusk suggested that the elephant was born around 1980, while that of the root suggested death around 1994, a lifespan of at least 14 years, rather shorter period than the average lifetime of an elephant (ca. 80 years). In addition, the F<sup>14</sup>C values of fragments collected along a cross-sectional line suggested that the outer part of the tusk formed first with inner parts being deposited gradually with growth.

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#### 1. Introduction

In East Asia, personal seals made of animal tusk, stone, or wood, are widely used, and among these materials, ivory is considered to have the highest value. However, in 1973, the Washington Convention (Convention on International Trading in Endangered Species of Wild Fauna and Flora: CITES) (http://www.cites.org/) gave rise to global prohibition on the trading of endangered species of wild fauna and flora, both living and dead as well as any products made thereof. Elephants were among the endangered species that became protected by the convention. Japan entered into CITES in 1980, prohibiting international trading of elephant tusk and its products from/into Japan, except those items purchased prior to 1980. Use of radiocarbon (<sup>14</sup>C) contents to determine the age of ivory has therefore become important in trade to verify that a given ivory product was not harvested later than 1980, if no official documentation exists to prove the age of the sample.

Atmospheric concentrations of <sup>14</sup>C produced by nuclear bomb tests in the atmosphere between 1945 and 1963 show unique temporal variations from 1955 to present, slightly dependent on spatial location (according to proximity of nuclear test sites) [1]. These secular variations can be successfully used in age

estimations of natural carbonaceous materials formed by incorporating atmospheric  $CO_2$  during this time frame, and a few applications have been done to elephant tusk samples [2,3]. Here, we attempted to estimate ages based on temporal variations in bomb-produced-<sup>14</sup>C contents of a full elephant tusk. Thirty tusk fragment samples were used for <sup>14</sup>C analysis by AMS, resulting in estimations of the formation ages of different parts according to catalogued global <sup>14</sup>C contents [1].

#### 2. Sample material

A full left elephant tusk registered at Kyushu University (Lab No. 00LX25, sample No. 352; mass 32 kg) was used for analysis (Fig. 1a). The tusk was sectioned in advance into 6 pieces (Sec. 352–1 to 352–6) from the tip to the root, as shown in Fig. 2b. In the center of the tusk, a cavity can be observed, which is largest at the root end and gradually narrows along the tusk, almost closing toward the tip (Fig. 2b). Mitochondrial DNA analysis suggested that the tusk is a haplotype L3 belonging to the West African glade [4]. The exact geographical location of the elephant could not be identified; however, it is likely to have been raised in a forest environment, based on  $\delta^{13}$ C and  $\delta^{14}$ N analysis [5].

The structure of the elephant tusk is illustrated schematically in Fig. 2a and b. The left tusk measured 175.1 cm in length and approximately 13.8 cm in diameter at the root (Fig. 2a). The central

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**Fig. 1.** Photo of an elephant tusk analyzed for  $^{14}$ C. (a) Full view of the elephant tusk (No. 352) prior to  $^{14}$ C analysis. (b) The tusk sectioned into 6 pieces, showing Sec. 352–6, -5, -4, -3 and -2. A clear cavity can be seen in Sec. 352–6 and -5.



**Fig. 2.** Schematic illustration of the elephant tusk used for growth process analysis. The tusk was sectioned into 6 pieces and fragments in a block collected for <sup>14</sup>C analysis. (a) Sampling positions on the two root pieces are shown. Three samples (No. 17 to 15 from outer to inner) from Line-1, 175 cm from the tip of the tusk; 2 samples each (No. 14 to13 and No. 12 to 11) from Line-2 and Line-3, 161 and 146 cm from the tip; and 10 samples (No. 1 to 10) from Line-4, 133 cm from the tip of the tusk, were collected, as summarized in Table 1. (b) Blocks of tusk collected for <sup>14</sup>C analysis: from the top 4 sectioned pieces (Sec. 352–4, -3, -2, -1). The samples are listed in Table 1.

cavity located within the center of the tusk almost closed at a depth of around 70 cm from the root, leaving a dark tube (Fig. 2a and b). A dark zone on the surface of the tusk at approximately 45 cm from the root end was identified as staining from tannin, probably arising from food, suggesting that the tip from a boundary of approximately 130 cm was exposed from the body. The cross-section of the tusk was oval rather than circular in shape, with a relatively wider vertical diameter.

#### 3. Method

The six sections were further cut into two halves vertically and thin surface skin near the location of sample collection was removed and cleaned with a dental drill. Small tusk blocks were sampled from 30 positions as shown in Fig. 2a and b, using a wire-cutting tool at the machine factory of Nagoya University, and a dental drill equipped with a saw. The tusk fragment samples were collected in block. We did not extract collagen fraction from the tusk, as is normally used for <sup>14</sup>C dating of bone and tusk samples. Instead, we used the original block for combustion to collect CO<sub>2</sub>. We had already performed experiments of comparing the <sup>14</sup>C results for collagen and for blocks from the modern or well-preserved tusks. The <sup>14</sup>C results for the two fractions were consistent with each other within errors [6]. Thus we have concluded that the direct combustion of ivory gives us <sup>14</sup>C concentration that is consistent with that obtained by the collagen extraction method, provided that the ivory samples are of modern origin or preserved well in a specific condition [6]. The block samples were rinsed with acetone for a few days and then processed by acid-alkali-acid treatment at room temperature, treating successively with 0.6 M HCl for two hours, with 0.6 M NaOH for 3 h, and with 0.6 M HCl for 4 h. During the treatment with 0.6 M HCl. a weak bubble formation on the tusk block surface was recognized. Then the samples were rinsed with distilled water. and dried at 50 °C. After the treatments, the weights of samples decreased to 25-35% of their original values. The pre-treated samples were then combusted to  $CO_2$  [6–7]. The  $CO_2$  was separated and purified with a glass-vacuum system and reduced to graphite on an iron catalyst under a hydrogen atmosphere [6,7]. All 3 carbon isotopes of the graphite materials prepared from the samples, NIST HOxII standard and <sup>14</sup>C blank material (oxalic acid (No. 57952) synthesized from fossil-fuel carbon, available from Kishida Chemical Co. Ltd., Japan) were measured with an accelerator mass spectrometry (AMS) system (HVE model-4130) at Nagoya University [7–8]. The resulting  ${}^{14}C/{}^{12}C$  and  ${}^{13}C/{}^{12}C$  ratios were then used to calculate the  ${}^{14}C$  contents of the samples as follows:  $F^{14}C = (1.0/0.7459) \times [({}^{14}C/{}^{12}C)_{spl}/({}^{14}C/{}^{12}C)_{HOXII}]$ , with correction for carbon isotopic fractionation [9,10] (Table 1). The obtained F<sup>14</sup>C values were calibrated to calendar dates using the CALIBomb program (14C-chrono program) developed by the Center for Climate, the Environment, and Chronology at Queen's University in Belfast, Ireland (http://www.calib.gub.ac.uk/CALIBomb/). For age calibration, we used <sup>14</sup>C temporal variations given by southern-hemisphere zone-3 (SHZ3) compilation. because it is considered that the tusk analyzed was from an elephant grown up in West Africa, and most probably come from the Republic of the Congo. Anyway, the <sup>14</sup>C variations of SHZ3 are almost identical to those of northern-hemisphere zone-3 (NHZ3) [1]. Atmospheric <sup>14</sup>C curves used for calendar age calibration of F<sup>14</sup>C value form a shape of convex upward. Namely, there are two possible calendar ages for each measured F<sup>14</sup>C value. It is natural and in fact proved by experiments [2,3] that the tip of tusk formed first and the base of the tusk later on. According to this constraint, all our measured F<sup>14</sup>C values on a full elephant tusk were calibrated to calendar ages consistently by using the decreasing part of atmospheric <sup>14</sup>C curve. The estimated formation age of each sample is given in Table 1, along with  $\delta^{13}$ C values with errors of ±ca. 1.0‰, measured with the AMS system.

#### 4. Results and discussion

#### 4.1. $\delta^{13}C$ values

 $\delta^{13}$ C values measured by AMS ranged from -25% to -28% (Table 1), suggesting that the elephant survived mainly on a diet

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