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Preliminary Dating of a Hominid Fossil Tibia from Sambungmacan by Fluorine Analysis*

By

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Abstract In September 1977, part of a well-fossilized tibial shaft of man was found together with faunal remains from the Solo River short-cut canal site at Sambungmacan in Central Java, Indonesia. The sediments exposed at the site have been tentatively divided into 4 units; Unit-Sb1, Unit-Sb2, Unit-Sb3 (late Pleistocene terrace deposits) and Unit-Sb4 (recent alluvial deposits) in ascending order: Unit-Sb1 and Unit-Sb2 have been inferred to be correlative with the Kalibeng and the Kabuh Formation in the Sangiran area respectively. While the exact findspot of the human tibia (surface discovery) in the field is unknown, the fluorine content of this specimen compares well with those of vertebrate bones *in situ* from the basal to the lower part of Unit-Sb2. Provided that the tibia derives from this Kabuh equivalent, it should be comparable in age to early Javanese fossil hominids. However, some other layers are still possible for the source of the tibia. Further stratigraphic and chronological investigations are required to clarify the provenances of the Sambungmacan hominid remains including the calotte found in 1973 with characteristics of Solo man skulls.

Introduction

An adult calotte of a fossil hominid (e. g. JACOB, 1981) was encountered in February 1973 near the Sambungmacan village, Central Java (Fig. 1), in the course of construction of a short-cut canal at a meander site of the Solo River. In September 1977 an intensive field survey was carried out at Sambungmacan by the Indonesia-Japan joint research team in the framework of the Cooperation Technical Assistance Project Number 41 called CTA-41 (see WATANABE & KADAR [eds.], 1985), to make a detailed geological map of the sediments exposed along the canal (SHIBASAKI *et al.*, 1985) and to locate the source horizon of the 1973 skull cap (registration number: Sambungmacan 1 or Sm 1).

Fossil vertebrate remains were also collected during this 1977 fieldwork, and later found included in the Sambungmacan collection was a hominid tibial fragment

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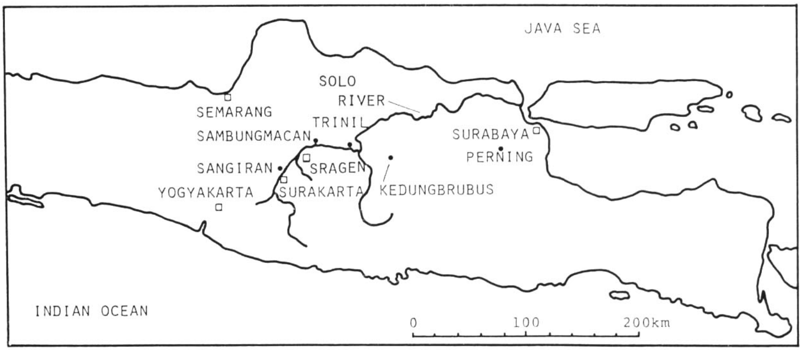

Fig. 1. Central and East Java showing some fossil hominid sites (small dots) and towns (open squares).

(see BABA *et al.*, 1990).

This hominid specimen, having not been recovered *in situ*, lacks individual record of the exact location of it in the canal exposure. Still, it may presume a great antiquity of the tibia that the bone is black-coloured throughout to the internal portion of cortex and heavily petrified.

While the stratigraphic reexamination of the Sambungmacan site (Indonesia-Japan Joint Study Team, 1989) and the morphological study of the hominid tibia (BABA & AZIZ, 1989; BABA *et al.*, 1990) were undertaken, the present research was initiated to deduce the original stratigraphic provenances of the Sambungmacan hominid remains and assist the relative dating of them by some geochemical approach. This paper is a note on the application of the fluorine dating method (see OAKLEY, 1951, 1980; MATSU'URA, 1982, 1986 a) to the Sambungmacan tibia; this involves assay of the specimen in question together with vertebrate fossils collected *in situ* from the same site, and also comparison of the analytical results obtained with series of fluorine data on bones from other two Pleistocene hominid sites, Sangiran and Trinil (MATSU'URA, 1982, 1986 b), in the drainage basin of the Solo River (Fig. 1).

Material

Vertebrate fossils collected from the Solo River short-cut canal site at Sambungmacan in September 1977 by the CTA-41 Indonesia-Japan Joint Research Project Team (see WATANABE & KADAR, [eds.], 1985), were submitted to this analytical study. The number of the bones used and their geological contexts are as follows (see Table 1 & Fig. 2).

Animal

Eleven specimens are *in situ* from the consolidated sand and gravel bed exposed at the canal bottom, and nine specimens *in situ* from a gravel bed at the north-wall of

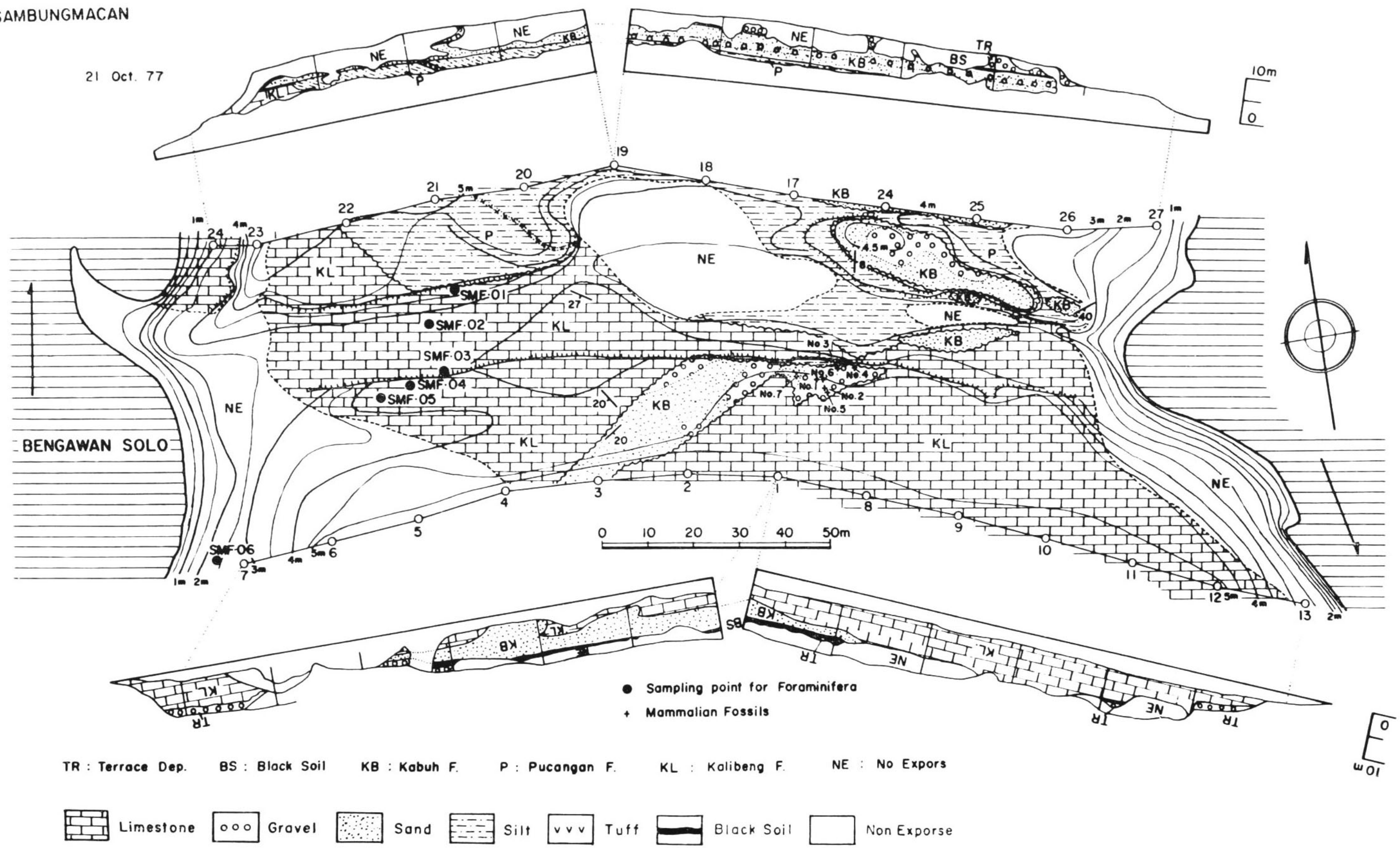

Fig. 2. Geological development of the Sambungmacan short-cut site (after Indonesia-Japan Research Cooperation Programme—CTA-41, 1979). The Pucangan Formation identified here is now assigned to the lower part of the Unit-Sb2 (eq. to the Kabuh Formation, see Table 1).

Table 1. Tentative stratigraphical correlation of the Sangiran area and the Solo River bank exposures near Sragen (after Indonesia-Japan Joint Study Team, 1989). This Solo River area includes Sambungmacan.

Sangiran	Solo River bank near Sragen	
	Summarized	Sambungmacan
Alluvium	Alluvium	Unit-Sb4
Terrace deposits	Terrace deposits	Unit-Sb3
Pohjajar F. (Notopuro F.)	Unit-S4	—
Bapang F. (Kabuh F.)	Unit-S3	Unit-Sb2
Sangiran F. (Pucangan F.)	Unit-S2	—
Puren F. (Kalibeng F.)	Unit-S1	Unit-Sb1

F. = Formation

the canal.

These two fossil-bearing horizons lie in the basal to lower parts of Unit-Sb2 of the strata developed at the site. There has been discrepancy between views of the stratigraphic sequence of Sambungmacan (Indonesia-Japan Research Cooperation Program—CTA-41, 1979; SARTONO, 1979; BARTSTRA, 1982; and others: for this problem, see SHIBASAKI *et al.*, 1985). However, recent geological resurvey infers that Unit-Sb2 is correlative with the Kabuh Formation in the Sangiran area which affords a fairly well-dated stratigraphic succession as a useful correlation standard (Indonesia-Japan Joint Study Team, 1989).

Besides, a surface find of bone bearing a close resemblance in color and petrification to the human tibia was also used for a preliminary experiment (see the next Method section).

Human

The bone is about 10 cm long, and referable to the lower part of right tibial shaft (BABA & AZIZ, 1989; BABA *et al.*, 1990). This specimen had been inscribed 'SB210977' that means 'Sambungmacan, 21st September in 1977', but the exact findspot is unknown.

The registration number of Sambungmacan 2 (or Sm 2) will be assigned to this tibia (see BABA *et al.*, 1990).

Method of Fluorine Analysis

Sample Preparation

A small transverse section of *substantia compacta* (or *dentinum*) was cut from each bone specimen, and cleaned to remove surface dirt or other adhering matter. The sample was then ground to fine, homogeneous powder with an agate mortar and pestle.

In the case of the hominid tibia mentioned above, approximately 0.05 g of powder was obtained from an internal portion of cortical bone using a dental drill, after the medullary surface at the drilling spot was scraped away to eliminate possible contaminants such as foreign mineral grains and soli particles. The powder was further pulverized in a mortar. It may be remarked here that this tibial bone powder was not so prepared from a section extending from the outer to the inner surface of compact tissue as is usually the case with animal specimens. Accordingly, the effect of zonation of fluorine, that is, variation in fluorine content with depth from the exposed surfaces of compact bone, should receive consideration. Yet, such variation is known to be minor for Pleistocene fossil remains of Sangiran in Central Java (MATSU'URA, 1982), and a preliminary experiment has shown that the tendency of the depth-dependent variation being minor may be applicable to the case of Sambungmacan remains involved here. A more extensive discussion of the care to be taken and problems encountered on applying fluorine dating procedures, will be found in MATSU'URA (1982).

Apparatus

An Orion Model 94–09 fluoride ion-selective eletrode was employed in conjunction with a Corning Model 250 digital (0.1 mV resolution) pH/ion analyzer, or an Iwaki Model 008F fluoride electrode with a Corning Model 130 digital (0.1 mV resolution) pH/ion meter. An Iwaki Model 109 calomel electrode filled with 4M KCl was used as reference.

Reagents

All chemicals were of guaranteed reagent grade or ultra-fine grade.

Buffer Solution

To about 350 ml of distilled water was added 250 ml of 1 M KOH, 147 g of sodium citrate dihydrate and 10.2 g of sodium acetate trihydrate. The solution was allowed to warm to room temperature and diluted to 1 liter with distilled water.

Sodium Fluoride Standards

The NaF standard solution from TOA Electronics that contains 1000 ppm (mg per liter) F^- was diluted as required. At least two standards are required which bracket the expected fluoride range of bone sample solutions and differ in concentration by a factor of approximately 10. In this experiment 10, 40 and 100 ppm F^- standards were prepared.

Procedure

Ten to 20 mg of powdered bone sample was weighed out to an accuracy of 0.05 mg in a 30-ml beaker, dissolved by addition of 5 ml of 1 M HCl, and 5 ml of distilled water was added. Then 10 ml of the buffer solution was mixed to provide a constant background ionic strength, dissociate fluoride complexes and adjust the pH of the sample solution. The buffered system has final concentrations of 0.125 M HCl, 0.125 M KCl, 0.0375 M NaAcetate and 0.25 M NaCitrate. The beaker was covered tightly with a self-sealing film. Standard solutions for calibration with fluoride concentrations of 2.5, 10 and 25 ppm as F^- may be prepared by substituting 5 ml of the respective NaF standards containing 10, 40 and 100 ppm F^- for the 5 ml distilled water, along with hydroxyapatite powder (see MATSU'URA, 1981) for the bone sample. The dissolved amount of hydroxyapatite should approximate the average amount of bone samples weighed out in a series of measurements.

The sample and standard solutions were allowed to stand for about 1 hour to come to the same temperature. The two (fluoride & calomel) electrodes, having been conditioned before use, were immersed into the solution which was stirred with a Teflon-coated magnetic bar, and the millivoltage reading in equilibrium was recorded. The temperature of the solution was checked to be within ± 0.2°C of each other.

The fluoride concentration of the sample solution was determined by reference to a direct calibration curve (here a straight line) obtained with data on the standard solutions; this was further converted into the percent fluorine content of the bone sample from the mass of the dissolved bone powder. It should be noted that calibration is required for every series of potential measurements, generally 10 samples involved. The direct measurement results were occasionally verified by known addition procedure.

A general discussion and a detailed description of the electrode method for fluorine analysis of bone are available in MATSU'URA (1981) and MATSU'URA (n. d.) respectively.

Results and Discussion

Analysis of the compact tissue sample of the human tibial fragment from Sambungmacan yielded 2.37% F. The results of fluorine test on the animal bone specimens are included in Tables 2 & 3. The reproducibility (represented by C. V.) of the fluorine determination is 0.010 to 0.013 (see MATSU'URA, n. d.); this implies for instance that the precision is to be about ± 0.03% F (1σ) when the measurement result is 2.50% F.

Phosphate was also determined on some of the bone powder samples by normal volumetry according to the procedure described in MATSU'URA (1982). Phosphate determination gives a convenient measure of bone apatite content, and serves to examine the possibility of the fossil bone sample being contaminated by infiltrated minerals or other adventitious substances that should correspondingly cause an apparent lowering in fluorine content of the bone. The analytical results (Tables 2 & 3), on referring to reported P_2O_5 data for fossilized bones and teeth (e. g. MATSU'URA, 1982, 1986 b), demonstrate that these samples have suffered little or no significant contamination from extraneous matter, and that the fluorine content values of the Sambungmacan specimens used in this study would not need particular corrections.

MATSU'URA (1982) measured fluorine on a large number of vertebrate fossils excavated from the Sangiran anticline area to obtain a series of standard data for tracing back the provenances or checking the reported/alleged source beds of the Sang-

Table 2. Analyses of fluorine and phosphate in compact bone of fossil specimens *in situ* from the bottom exposure of the short-cut canal at Sambungmacan. Horizon: consolidated sand and gravel bed in Unit-Sb2 (cor. with the Kabuh Formation).

Specimen	Thickness of analysed part* (mm)	Fluorine %	P_2O_5 %	$100F/P_2O_5$ ratio
vertebrate, long bone	6.5	1.65	30.1	5.48
vertebrate, long bone	3.9	1.93		
vertebrate	4.0	1.98		
vertebrate	5.4	2.02	30.8	6.56
vertebrate	c. 3.3	2.02		
vertebrate, long bone	8.7	2.06		
vertebrate	6.0	2.20	33.4	6.59
mammal, rib	5.1	2.21		
mammal, rib	6.5	2.31		
vertebrate	c. 7.3	2.32		
vertebrate, long bone	3.5	2.66	33.5	7.94
	Mean	2.12		
	S. D.	0.26		

* Thickness of compact tissue.

Table 3. Analyses of fluorine and phosphate in compact bone and horn and in dentine of fossil specimens *in situ* from the north wall exposure of the short-cut canal at Sambungmacan. Horizon: gravel bed in Unit-Sb2 (cor. with the Kabuh Formation).

Specimen	Thickness of analysed part* (mm)	Fluorine %	P_2O_5 %	$100F/P_2O_5$ ratio
vertebrate, long bone	9.5	1.99	34.3	5.80
vertebrate, long bone	c. 6.1	2.10		
vertebrate	>6.1	2.27		
vertebrate, long bone	6.1	2.31	34.4	6.72
vertebrate, tooth	>4.4	2.33	32.1	7.26
mammal, horn	>6.0	2.35		
mammal, tooth	>6.3	2.52		
vertebrate	4.4	2.59	33.6	7.71
vertebrate	4.0	2.84		
	Mean	2.37		
	S. D.	0.26		

* Thickness of compact tissue or dentine.

iran hominids. As Fig. 3 shows, the content of this element in bones from different stratigraphic levels varies, reflecting the different lithologic circumstances of burial media. The fluorine measurements on the Sambungmacan material (Tables 2 & 3) are compared hereunder with those of faunal remains from Sangiran and also from Trinil (MATSU'URA, 1986 b).

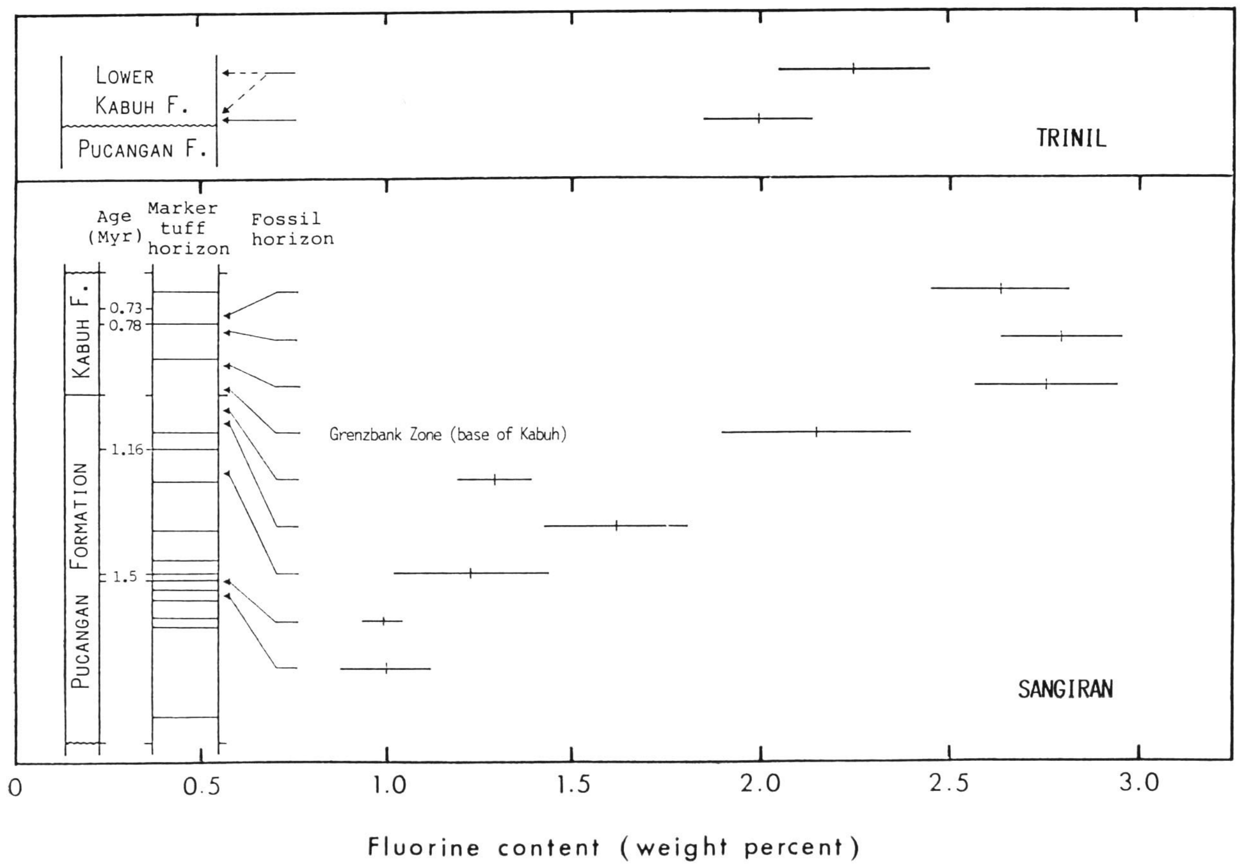

Fig. 3. Fluorine content (mean±S. D.) of *substantia compacta* or *dentinum* of fossil vertebrate remains from the Trinil area (based on MATSU'URA, 1986 b) and the Sangiran area (based on MATSU'URA, 1982) in Java. The stratigraphical contexts and absolute ages depend on the work by the CTA-41 team (WATANABE & KADAR [eds.], 1985). Recent palaeomagnetic study (HYODO *et al.*, 1988) suggests age estimates of c. 0.9 Myr for the Kabuh/Pucangan boundary and c. 1.7 Myr for the Pucangan/Kalibeng boundary in the Sangiran area.

The fossil bones from the basal to lower parts of the Kabuh equivalent layers at the Sambungmacan canal site show the average fluorine content of 2.12% (S. D.= 0.26%, n=11; Table 2) for the consolidated sand and gravel bed, and of 2.37% (S. D.=0.26%, n=9; Table 3) for the gravel bed: these values are found to be similar to that for fossil bones from the calcareous conglomeratic sandstone bed (so-called Grenzbank zone) lying at the base of the Kabuh Formation of the Sangiran area (see Fig. 3). In particular, bones from the consolidated sand and gravel bed approximate those from the Grenzbank zone in fluorine content. This good approximation corresponds well to the resemblance of facies between the two fossil-bearing beds involved (see WATANABE & KADAR [eds.], 1985). The Sangiran specimens from the Kabuh Formation exclusive of Grenzbank (Fig. 3) have a tendency to contain higher fluorine than the Sambungmacan faunal specimens, although the fluorine measurements on the former (MATSU'URA, 1982) overlap more or less with those given in Table 2 or Table 3.

Turning to the case of Trinil, fossil remains from the basal (or the lower in part?) Kabuh are comparable in fluorine content (Fig. 3) to the Sambungmacan bones from the basal to lower 'Kabuh' (Tables 2 & 3), as could have been expected.

These comparisons presented above are outlined in Table 4, which also represents available data relevant to allowing an inference of the stratigraphic position of the hominid tibia from Sambungmacan to be made. The fluorine content of the hominid specimen, falling within the range of fossils from the basal to lower parts of Unit-Sb2, tentatively suggests that the Sambungmacan tibia has its origin in the Kabuh equivalent layers. Then it would be supposed to be referred to early Javanese hominids such as pithecanthropines (Java man).

It should be noted here, however, that the skull cap (Sambungmacan 1) found in 1973 has characteristics of Solo man that is represented by the Ngandong skulls

Table 4. Summarized fluorine data (range and mean) on vertebrate remains from the Kabuh Formation and its equivalent, in reference to the Sambungmacan tibia.

Sambungmacan (this study)	Sangiran (MATSU'URA, 1982)	Trinil (MATSU'URA, 1986b)
Unit-Sb2 (Kabuh equivalent) consolidated sand and gravel bed at bottom exposure of canal: 1.65 to 2.66 %F (Mean 2.12 %F) Unit-Sb2 (Kabuh equivalent) gravel bed at north wall exposure of canal: 1.99 to 2.84 %F (Mean 2.37 %F) human tibia 2.37 %F	Kabuh Formation basal part (Grenzbank): 1.65 to 2.77 %F [mainly 1.8 to 2.4 %F] (Mean 2.15 %F) Kabuh Formation excluding Grenzbank: 2.31 to 3.16 %F (Mean ca. 2.7 %F)	Kabuh Formation basal part: 1.75 to 2.21 %F (Mean 1.99 %F) Kabuh Formation basal part or somewhat upper horizon in lower part: 1.96 to 2.50%F (Mean 2.25 %F)

of much later age than Java man. Future researches are necessary to clarify the source horizons of the Sambungmacan hominid specimens and to establish the stratigraphical sequence of the site.

Reinvestigations on the Quaternary deposits in Central and East Java are currently in progress that involve collecting vertebrate fossils. Continuing studies, analysing bone specimens for fluorine and other elements of geological interest, are fundamental to the construction of the chronology, relative or absolute, for the fossil hominids of Java.

Note Added in Proof

The latest field survey in 1990 by the Indonesia-Japan Joint Research Team has suggested that layers which may be correlated to the Setri Formation of the Sawur River area (see Indonesia-Japan Joint Study Team, 1989) can be distinguished at the Sambungmacan site. These 'Setri' layers of Sambungmacan overlie 'Kabuh' layers and contain mammalian fossils. The possibility that the Sambungmacan tibia (and also the 1973 calotte) comes from the 'Setri' part should be recognized, and is to be the subject of extended examinations. In any case, the Sambungmacan tibia would be assigned to a fossil hominid which may be included in Solo man or Java man.

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