F. McDermott
Department of Geology, University College Dublin, Belfield, Dublin 4, Ireland

C. Stringer
Department of Palaeontology, The Natural History Museum, London SW7 5BD, U.K.

R. Grün
Quaternary Dating Research Centre, Australian National University, Canberra, A.C.T. 0200, Australia

C. T. Williams &
V. K. Din
Department of Mineralogy, The Natural History Museum, London SW7 5BD, U.K.

C. J. Hawkesworth
Department of Earth Sciences, The Open University, Walton Hall, Milton Keynes, MK7 6AA, U.K.

Received 7 May 1996
Revision received 22 August 1996 and accepted 25 August 1996

Keywords: Singa, hominid, U–Th dating, ESR dating, U–Th isochrons.

New Late-Pleistocene uranium–thorium and ESR dates for the Singa hominid (Sudan)

The Singa (Sudan) calvaria has been interpreted previously as a terminal Pleistocene modern human fossil, perhaps related to the Bushman of Southern Africa. Here we report new mass-spectrometric U–Th dates for the calcrete deposit enclosing the fossil teeth and the calvaria itself and new electron spin resonance (ESR) dates for associated dental materials. The new data constrain the age of the hominid to at least 133 ± 2 ka. Together with the preferred linear uptake (LU) ESR dates, the U–Th data confirm that the intriguing mixture of modern and archaic characteristics in the Singa specimen date from isotope stage 6. Far from being a modern human fossil, it represents a rare example of an archaic African population which may have been ancestral to all modern *Homo sapiens*.

Journal of Human Evolution (1996) **31,** 507–516

Introduction

Morphological and genetic evidence suggest that *Homo sapiens* originated in Africa some 200–130 ka ago (Grün & Stringer, 1991; Horai *et al.*, 1995; Nei, 1995; Goldstein *et al.*, 1995), but these critically important African populations remain poorly known and dated (Grün & Stringer, 1991; Smith, 1993). A well-preserved hominid fossil from Singa, Sudan, previously supposed to represent a modern specimen (Rightmire, 1984; Clark, 1988) may represent such a population (Grün & Stringer, 1991). The Singa fossil was discovered in 1924 by Bond, eroding out of a calcrete deposit in the west bank of the Blue Nile, some 320 km south east of Khartoum (Woodward, 1938). Mammalian fauna and Palaeolithic artefacts collected from the locality and from Abu Hugar, some 15 km to the south (Arkell *et al.*, 1951) yielded extinct, possibly Upper Pleistocene fauna (Bate, 1951). Prior to 1990, a radiocarbon date of ca. 17 ka on a crocodile tooth from Abu Hugar (Whiteman, 1971) was the only absolute date available. Thus, the Singa hominid was regarded as "proto-Bushman" by Woodward (1938), and since then it has been attributed to modern *H. sapiens* (Rightmire, 1984; Clark, 1988). However, a minority (e.g. Tobias, 1968; Brothwell, 1974; Stringer, 1979) have emphasized its archaic

0047–2484/96/120507+10 $25.00/0

morphological and metrical features, and further study has supported Brothwell's view that the unusual shape of the parietals might be related to pathology (Stringer *et al.*, 1985). Preliminary electron spin resonance (ESR) dates on an associated *Equus* and bovid tooth from the Singa site (Grün & Stringer, 1991) had suggested an oxygen isotope stage 5–6 age, but these dates remained equivocal due to large discrepancies between early (EU) and linear (LU) uranium uptake models, and the uncertain contemporaneity of the dated teeth and the calvaria. Here we present new mass-spectrometric U–Th dates for the calcrete matrix found adhering to the external surface of the calvaria and enclosing associated mammal teeth. Unlike dental materials (e.g. enamel, dentine) which acquire uranium subsequent to burial, calcretes incorporate uranium into the calcite lattice at the time of deposition, thereby eliminating a major source of uncertainty which hinders U–Th and ESR dating of fossil teeth. Calcrete deposition clearly post-dated the burial of the fossil material and deposition may have continued over a period of time, implying that the U–Th dates reported here are minimum estimates for the age of the Singa hominid.

Analytical techniques

The difficulties associated with dating detritally-contaminated carbonate material have been well-documented and they arise primarily because, depending on experimental conditions, the detritus may contribute variable and unpredictable amounts of ^{232}Th, ^{230}Th, ^{238}U and ^{234}U to the sample solution (e.g. Schwarcz & Latham, 1989; Bischoff & Fitzpatrick, 1991; Luo & Ku, 1991; Kauffman, 1993). In this study, U and Th isotope measurements were carried out on acid leachates of calcrete, and were used to construct a mixing line in three dimensional (^{230}U/^{232}Th), (^{234}U/^{232}Th), (^{238}U/^{232}Th) space, the slope of which yields the (^{234}U/^{238}U) and (^{230}Th/^{238}U) ratios of the pure carbonate end-member (Rosholt, 1976). Several impure calcrete samples with variable carbonate/silicate ratios were partially dissolved in teflon-distilled 2 M HNO_3, and all insoluble residues were removed by centrifuging. For comparison with ESR dates (Table 2) a single fossil equid tooth (596) was also analysed, and unlike the calcrete this was essentially detritus free and could be dissolved completely in 7 M HNO_3. Following aliquoting, spike addition (^{229}Th-^{235}U) and two-stage ion-exchange chromatography, the ^{234}U/^{238}U, ^{230}Th/^{229}Th and ^{232}Th/^{229}Th ratios in all samples were measured in ion-counting mode using a high-abundance sensitivity ($\sim$10 ppb), low darknoise (<0·03 cps) thermal ionization mass spectrometer at the Open University (MAT 262 RPQ-II). Uranium contents were determined on ^{235}U spiked aliquots at University College Dublin using a single collector mass spectrometer (MM30). Total procedural blanks were negligible at <20 pg for ^{232}Th and <5 pg for ^{238}U. All isochron age calculations employed a three-dimensional line-fitting procedure based upon the maximum likelihood estimation approach (Titterington & Halliday, 1979; Ludwig & Titterington, 1994) as implemented in v2.8 of the ISOPLOT program (Ludwig & Titterington, 1994). This procedure takes account of correlated errors, and the fact that both parts of the Rosholt-type isochron diagrams (Figure 1) share the same (^{238}U/^{232}Th) data. Isochron slope and age uncertainties are quoted at the 95% confidence level unless otherwise stated.

The calcrete sub-samples selected for acid leaching were 2–5 mm fragments (0·5–1·0 g) picked at random from heterogeneous lightly crushed bulk samples. U–Th isotope analyses were carried out on three acid-leachates of the calcrete matrix found adhering to the exterior of the skull (samples SS1/L1, SS1/L2, SS1/L3; hereafter referred to as the external matrices), along with a residue of one of these leachates (SS1/L1R). Leachates of the calcrete

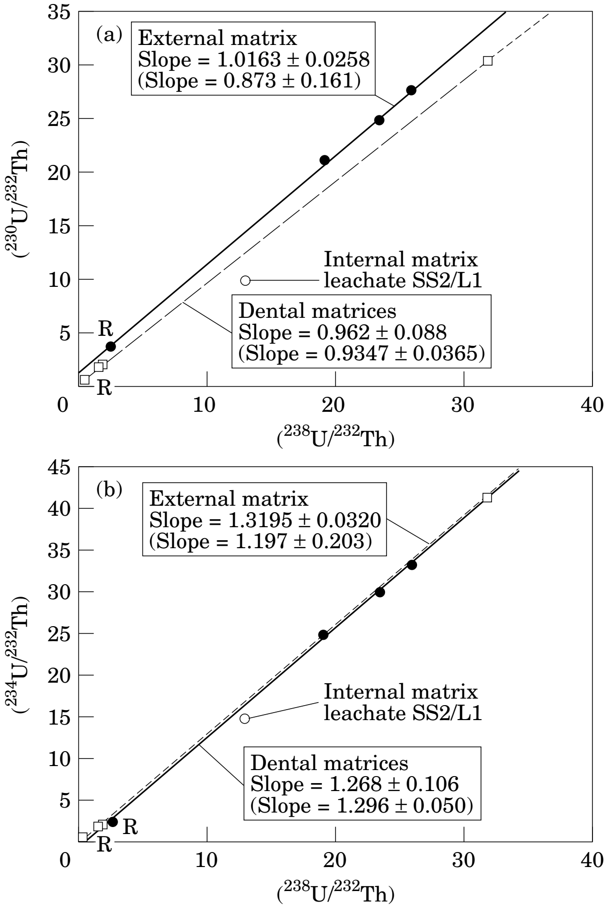

Figure 1. (a) (^{230}U/^{232}Th) against (^{238}U/^{232}Th) isochron plot for the Singa calcrete leachates and residues. Three leachates and one residue analysed from calcrete matrix found adhering to the exterior of the skull (filled circles) defines the upper (solid) line, and the dental matrices (open squares) and one associated residue define the lower (dashed) line. A 2σ relative error of ± 3% was assigned to each data-point to take account of a typical within-run 2σ relative error of 1·5%, and an allowance for the external variance observed in repeated analyses of a standard calcite sample (1·5% relative at the 2σ level). Error correlations were 0·1–0·2, reflecting the relatively precise ^{232}Th measurements. Slope errors are quoted at the 95% confidence level and are computed using a 3-D line fitting procedure (ISOPLOT v. 2.8, Ludwig & Titterington, 1994). Slope values in parentheses are for an isochron calculation based on leachates only (residue omitted). (b) (^{234}U/^{232}Th) *vs.* (^{238}U/^{232}Th) for the Singa calcrete leachates and leachates. Slope errors are quoted at the 95% confidence level as computed in Figure 1(a). Slope values in parentheses are for an isochron calculation based on leachates only (residue omitted).

matrix adhering to a fossil bovid tooth (SS3, Sample 597 of Table 2) and a fossil Kobus tooth (SS4) found in association with the calvaria (hereafter referred to as the dental matrices), and a residue from one of these leachates were also analysed (Table 1). In addition, one leachate of calcrete from the interior of the calvaria (sample SS2) and two sub-samples (dentine and enamel) from an *Equus* tooth associated with the Singa specimen were analysed (sample 596, Table 1). A separate sub-sample from the latter tooth was also dated by ESR (Table 2).

Table 1 Uranium–thorium isotope data for the Singa calcrete matrices and an associated *Equus* tooth

Sample	^{238}U μg/g	(^{234}U/^{238}U)	(^{230}Th/^{232}Th)	(^{230}Th/^{234}U)	(^{234}U/^{232}Th)	(^{238}U/^{232}Th)	Age (ka)
Singa calvaria external matrix							
SS1/L1	3·19 ± 0·04	1·274 ± 0·007	24·58 ± 0·05	0·821 ± 0·011	29·95 ± 0·20	23·513 ± 0·382	
SS1/L2	3·80 ± 0·04	1·275 ± 0·007	27·53 ± 0·07	0·829 ± 0·009	33·21 ± 0·23	26·040 ± 0·367	
SS1/L3	2·167 ± 0·008	1·300 ± 0·007	21·26 ± 0·29	0·854 ± 0·012	24·79 ± 0·11	19·062 ± 0·113	
SS1/L1R	0·90 ± 0·01	0·980 ± 0·007	3·71 ± 0·03	1·564 ± 0·013	2·375 ± 0·005	2·422 ± 0·005	
					External matrix leachate–residue isochron age=		145·5 ± 7·5
					External matrix leachate–leachate isochron age=		133 ± 26
Associated dental matrix							
SS3/L1R	0·79 ± 0·001	1·007 ± 0·007	0·776 ± 0·007	1·706 ± 0·001	0·455 ± 0·001	0·452 ± 0·001	
SS3/L2	2·454 ± 0·004	1·192 ± 0·008	1·771 ± 0·018	1·044 ± 0·002	1·69 ± 0·02	1·423 ± 0·018	
SS4/L1	0·612 ± 0·003	1·157 ± 0·008	2·07 ± 0·020	1·024 ± 0·009	2·017 ± 0·038	1·742 ± 0·039	
SS3/L1	9·888 ± 0·016	1·296 ± 0·008	30·28 ± 0·300	0·732 ± 0·005	41·35 ± 0·36	31·902 ± 0·264	133 ± 2*
					Dental matrix leachate–residue isochron age=		143 ± 27
					Dental matrix leachate–leachate isochron age=		129 ± 5
Singa skull interior matrix							
SS2/L1	0·441 ± 0·001	1·163 ± 0·002	9·734 ± 0·089	0·657 ± 0·009	14·81 ± 0·11	12·735 ± 0·071	—
Singa *Equus* tooth							
596DE	51·86 ± 0·18	1·3602 ± 0·007	280·19 ± 1·9	0·497 ± 0·001	564·12 ± 5·7	414·693 ± 2·83	72·01 ± 0·21
596EN	31·62 ± 0·29	1·3784 ± 0·007	385·54 ± 1·22	0·583 ± 0·005	661·77 ± 0·93	480·06 ± 3·59	90·29 ± 1·2

Parentheses denote activity ratios. Decay constants used to convert from measured atomic ratios to activity ratios were λ^{230}Th=9·195E-6, λ^{234}U=2·835E-6, λ^{238}U=1·551E-10,λ^{232}Th=4·948E-11. SS1 is a sample of the calcrete matrix found adhering to the exterior of the Singa calvaria. SS2 is a sample of the calcrete from the interior of the skull and samples SS3 and SS4 are samples of calcrete matrix found adhering to an associated bovid (sample 597) and *Kobus* tooth respectively. Sample numbers ending with L1, L2 etc. are acid leachates of different aliquots of samples SS1, SS2, SS3 etc. and those ending in R are residues (e.g. SS1/L1R is the residue from leachate SS1/L1). SS2/L1 is omitted from the isochron calculations (see text). Samples 596DE and 596EN are dentin and enamel respectively of the *Equus* tooth 596. Errors on ratios are quoted at the 1σ level and reflect only the within-run counting statistics error. *Leachate SS3/L1 has a high (^{230}Th/^{232}Th) indicating negligible contamination by non-carbonate detritus. This allows calculation of a robust isochron-independent U–Th age for this sample (133 ± 2 ka).

Table 2 Results of chemical analysis and ESR age estimates for samples from Singa

Sample no	D_E (Gy)	U(EN) (ppm)	U(DE) (ppm)	TT (μm)	S1/S2 (μm)	Sediment U (ppm)	Sediment Th (ppm)	Sediment K (%)	Sediment γ-$\dot{D}$ (γGy/a)	Sediment β-$\dot{D}$ (μGy/a)	Early U-uptake int.$\dot{D}$ (μGy/a)	Early U-uptake De-$\dot{D}$ (μGy/a)	Early U-uptake Total $\dot{D}$ (μGy/a)	Early U-uptake Age (ka)	Linear U-uptake int.$\dot{D}$ (μGy/a)	Linear U-uptake De-$\dot{D}$ (μGy/a)	Linear U-uptake Total $\dot{D}$ (μGy/a)	Linear U-uptake Age (ka)
596a	293 ± 23	5·48	46·8	860	25	4·8	1·6	0·31	476 ± 83	205 ± 17	1607 ± 197	1005 ± 79	3293 ± 229	88·9 ± 9·3	819 ± 104	508 ± 40	2008 ± 140	146 ± 15
596b	306 ± 14	3·06	66·4	800	25	4·8	1·6	0·31	476 ± 83	213 ± 18	928 ± 122	1511 ± 114	3128 ± 187	97·8 ± 7·3	471 ± 62	762 ± 59	1922 ± 121	159 ± 12
596c	366 ± 24	5·85	73·7	1020	25	4·8	1·6	0·31	476 ± 83	186 ± 16	1777 ± 218	1476 ± 111	4906 ± 259	93·6 ± 8·7	913 ± 116	746 ± 58	2321 ± 155	157 ± 15
597a	234 ± 16	2·58	64·5	1150	25	5·5	1·6	0·10	476 ± 83	164 ± 14	778 ± 100	1188 ± 89	2606 ± 158	89·8 ± 8·3	392 ± 53	597 ± 46	1629 ± 110	144 ± 14
597b	277 ± 13	3·95	77·6	1100	25	5·5	1·6	0·10	476 ± 83	168 ± 14	1161 ± 145	1447 ± 107	3252 ± 199	85·1 ± 6·5	593 ± 77	733 ± 56	1970 ± 127	140 ± 11

EN, enamel; DE, dentine; TT, total enamel thickness; S1/S2, surface layer removed from each side of the enamel samples. Error in D_E after Grün & Brumby (1994); beta dose attenuation after Grün (1986); alpha efficiency, 0·15 ± 0·02; initial $^{234}U/^{238}U$, 1·42 ± 0·2 in enamel and dentin; water in dentin and sediment 10 ± 5 wt% (for beta dose-rate); average depth for cosmic dose rate, 5 ± 3 m (included in gamma dose-rate); neutron activation detection limits; U enamel and dentin, 0·1 ppm; Th and U in sediment, 0·1 ppm; K in sediment, 0·05 wt%.

Results

The four external matrix analyses (three leachates and one residue) define tight linear arrays on (^{230}Th/^{232}Th) *vs.* (^{238}U/^{232}Th) and (^{234}U/^{232}Th) *vs.* (^{238}U/^{232}Th) isochron diagrams (solid lines, Figure 1) and define slopes of $1{\cdot}0163 \pm 0{\cdot}0258$ and $1{\cdot}3195 \pm 0{\cdot}0320$ corresponding to the (^{230}Th/^{238}U) and (^{234}U/^{238}U) ratios respectively, of the pure carbonate component of the calcrete. These data yield an age of $145{\cdot}5 \pm 7{\cdot}5$ ka (MSWD = 1·15) at the 95% confidence level using the error limits and correlations outlined in Figure 1. To evaluate the extent to which the slope of the isochrons may be influenced by the low ratios of the residue data point (SS1/L1R, Table 1) and/or that the latter may have experienced differential isotope fractionation during the leaching step (e.g. Luo & Ku, 1991) we have also carried out an age calculation based only on the three leachates (residue omitted). This leachate-only calculation yields slopes of $0{\cdot}873 \pm 0{\cdot}161$ and $1{\cdot}197 \pm 0{\cdot}230$ for the (^{230}Th/^{238}U) and (^{234}U/^{238}U) ratios of the pure carbonate component (MSWD = 0·82), corresponding to an age of 134 ± 27 ka (± 54 ka at the 95% confidence level). Significantly, this age is within error of that defined by the four point (leachate–residue) isochron (see discussion below).

The four analyses of the dental matrix (three leachates and one residue) yield slopes of $0{\cdot}962 \pm 0{\cdot}088$ and $1{\cdot}268 \pm 0{\cdot}106$ (MSWD = 4·81) for the (^{230}Th/^{238}U) and (^{234}U/^{238}U) ratios, respectively, of the pure carbonate component (dashed lines, Figure 1). These data yield an age of 143 ± 27 ka at the 95% confidence level, and although the errors are larger than those of the external matrix, the age is indistinguishable from that defined by either the leachates alone (134 ± 27 ka) or the leachates plus residue for the external matrices ($145{\cdot}5 \pm 7{\cdot}5$ ka). In order to evaluate the extent to which the residue data point influences the dental matrix isochron we have also calculated an isochron age based only on the three leachates. This calculation yields slopes of $0{\cdot}9347 \pm 0{\cdot}0365$ and $1{\cdot}296 \pm 0{\cdot}050$ (MSWD = 1·73) for the (^{230}Th/^{238}U) and (^{234}U/^{238}U) ratios, respectively, of the pure carbonate component and corresponds to an age of 129 ± 5 ka ($\pm 9{\cdot}7$ at the 95% confidence limit). This result is within error of the leachate–residue (4 point) isochron indicating that in this case the residue does not unduly influence the slope of the leachate–residue isochron. The relative merits of leachate–residue *vs.* leachate–leachate isochrons are discussed below.

Significantly, one of the leachates (SS3/L1) has a high (^{230}Th/^{232}Th) ratio of ~ 30 (Table 1) indicating that it is essentially uncontaminated by silicate detritus and so it is possible to calculate a robust U–Th age for this single leachate. This yields an age of 133 ± 2 ka, well within the error envelope defined by the isochron on which it lies (143 ± 27 ka for the leachate–residue isochron or 129 ± 5 ka for the leachate only isochron). This strengthens considerably the argument for the antiquity of these deposits, because the age for SS3/L1 is entirely independent of any isochron calculation.

A single leachate analysed on material from the interior of the skull (SS2/L1) fails to lie on either isochron, indicating either that differential isotope fractionation occurred during the dissolution of this sample or perhaps more likely that its silicate (detrital) component differs from that of the other matrices.

Two sub-samples from an *Equus* tooth associated with the Singa specimen (sample 596, Table 1) were also U–Th dated to compare with ESR dates obtained for the same tooth (Table 2). Mechanical separation failed to produce a pure enamel sample for U–Th dating, and so while sample 596DE is comprised entirely of dentine, 596EN is impure and consists of dentine-contaminated enamel. Thus, both sub-samples are U-rich ($51{\cdot}86 \pm 0{\cdot}18$ and $31{\cdot}62 \pm 0{\cdot}29$ ppm, Table 1), and they yield U–Th ages of $72{\cdot}01 \pm 0{\cdot}21$ and $90{\cdot}29 \pm 1{\cdot}2$ ka

Table 3 **Results of combined ESR/U-series dating**

	Age	p(EN)	p(DE)
596A	94 ± 17	− 0·98 ± 0·14	− 0·77 ± 0·19
596B	127 ± 19	− 0·70 ± 0·15	− 0·41 ± 0·19
596C	110 ± 15	− 0·85 ± 0·12	− 0·59 ± 0·21

The parameter p(EN) and p(DE) is an uptake/diffusion parameter such that p= − 1 is a closed system and p= − 0 represents linear uptake (Grün & McDermott, 1994).

(1σ) respectively. ESR dates for enamel from sample 596 (Table 2) are in the range 89 ± 9·3 to 98 ± 7·3 ka if U-uptake occurred soon after burial (early uptake or EU model), or 146 ± 15 to 159 ± 12 if uranium uptake occurred through time in a linear fashion (LU model). A bovid tooth (597) was also dated by ESR and this yields an age similar to those of the *Equus* tooth (Table 2).

Like U–Th ages, ESR dates for fossil dental materials are sensitive to the sample's U-uptake history. In effect U–Th ages for fossil teeth are always calculated assuming an early uptake model, and they are similar, or somewhat younger than EU ESR ages (Table 2), as noted previously for Israeli sites (McDermott *et al.*, 1993). Thus, the U–Th and EU ESR ages on teeth are minimum ages, but the data can be combined to calculate model U–Th–ESR dates (Grün & McDermott, 1994) in the range 94 ± 17 to 127 ± 19 ka (Table 3).

In contrast with U–Th dates on dental materials, those for the calcrete are independent of the U-uptake history of coexisting teeth, and they provide a minimum age for the hominid enclosing sediments because calcite (calcrete) deposition would have post-dated sedimentation. Furthermore, the linear U-uptake ESR dates in the range 140 ± 11 to 159 ± 12 ka (Table 2) obtained for the associated fossil teeth are broadly consistent with the calcrete U–Th dates (ca. 130–145 ka), implying that the latter model closely approximates U-uptake history at this site.

Discussion

Calcrete is a relatively porous material and in principle it might be susceptible to open-system behaviour with respect to uranium. However, the fact that the U–Th analyses lie on tight well-defined isochrons testifies to the closed system behaviour of the calcrete at this site. Sample SS3/L1 is essentially free of any detrital contamination, and it provides a robust U–Th age of 133 ± 2 ka for the calcrete which enclosed the Singa calvaria. The remaining samples, by contrast, contain variable amounts of silicate detritus, and leachate–residue isochrons can be equivocal because of the possibility that the U-series nuclides in the residue may become fractionated during the sample dissolution step (e.g. Bischoff & Fitzpatrick, 1991). It is widely accepted however that U–Th isochrons based only on leachates (the so-called leachate–leachate or L/L methods of Schwarcz & Latham, 1989) are reliable, and these yield ages of 134 ± 27 ka (1σ) for the external matrix and 129 ± 5 ka (1σ) for the dental matrix. When the residues are included both isochrons appear to be biased slightly towards older ages (ca. 145 ka), but are still within error of the leachate-only isochrons. To circumvent the possibility, however remote, that the residues may have been affected by differential leaching

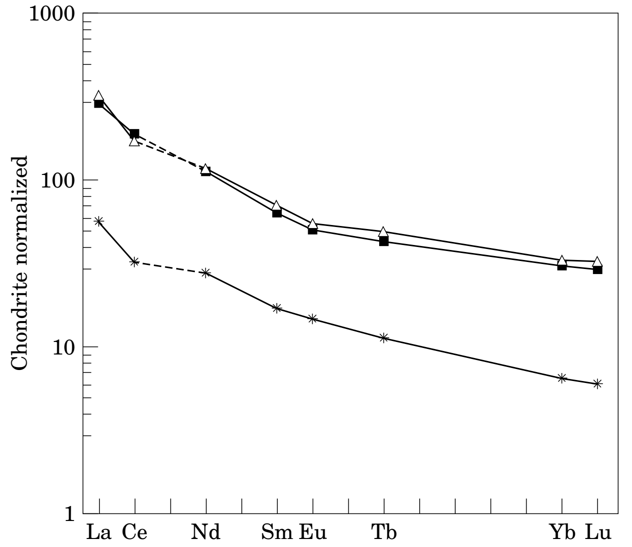

Figure 2. Chondrite normalized REE plots for calcrete found adhering to the *Equus* tooth [(■) 596], bovid tooth [(△) 597] and the interior of the calvaria [(*) 15546].

we present the leachate–leachate isochrons as our preferred age data. Thus, our preferred U–Th age for the calcrete, based on the result from sample SS3/L1 and the leachate–leachate isochrons is ca. 133 ka.

It is obvious that calcretes from different parts of the site have incorporated detrital material with different Th/U ratios (and hence different $^{230}Th/^{232}Th$ ratios). This observation is not unexpected because small variations in the mineralogy of the silicate detritus can have a detectable effect on its ($^{230}Th/^{232}Th$) ratio. Such differences can be resolved by the mass spectrometric technique used in this study, and they are responsible for the observation that the calcretes define a series of parallel isochrons. The division of the data into two isochrons is justified by the following arguments: (1) that local variations in the ($^{230}Th/^{232}Th$) of the detritus are expected and are consistent with the REE data (see below) (2) that both isochrons yield the same age (a geologically reasonable result) (3) that the leachates of both isochrons are collinear with analyses of their residues (4) that both (leachate–leachate) isochrons yield ages which are consistent with the U–Th age of the uncontaminated sample (SS3/L1). The alternative approach would have been to include all of the leachates (external and dental matrices) on a single isochron. Such an approach would be extremely difficult to justify in the light of (3) and (4) above. In any case such a combined leachate "isochron" would have steeper slope on Figure 1(a), and would yield an even older "age" of 155 ± 21 ka.

It is interesting to note that the EU and LU ESR results on several sub-samples of tooth 596 are in good agreement despite significant variations in all measured parameters (e.g. De-value, U concentrations, Table 2). When the U-series values of the dentine and enamel are used to establish combined U-series/ESR age estimates the results show a significant scatter (Table 3), presumably because the U concentrations analysed for the U–Th dating which were used in the combined age calculation do not adequately reflect heterogeneities on the scale of the sub-samples analysed for ESR. Nonetheless, the combined U–Th/ESR age estimates agree reasonably well with the U-series results.

Finally, the U–Th results are consistent with other geochemical variations within the calcrete. Calcrete from within the calvaria itself (15546) and adhering to the *Equus* (596) and bovid teeth (597) were analysed by inductively-coupled plasma emission spectroscopy (ICPES) and instrumental neutron activation analysis (INAA). The dental matrix samples (596 and 597) exhibit very similar normalized rare earth element (REE) contents and patterns [chondrite normalized lanthanum/ytterbium ratio (La_n/Yb_n) is ~5·7, Figure 2], indicating that they share a common detrital component, and by inference share a similar detrital ($^{230}Th/^{232}Th$) as implied by our treatment of the isochron data. However, the internal matrix (15546) has significantly lower REE concentrations consistent with REE dilution, but it also has a higher La_n/Yb_n ~7·9 indicating heavy REE depletion. Such compositional differences between the internal calcrete matrix and the dental matrices highlight spatial variations in the nature of the detrital component in different parts of the site, and are also consistent with the observation that the internal matrix does not lie on the dental matrix isochron of Figure 1.

Summary

In summary, the new U–Th dates for the calcrete deposits enclosing the Singa hominid indicate that it is at least 133 ± 2 ka old, and comparisons between the U–Th and ESR ages indicate a firm preference for a linear uptake uranium model at this site. The intriguing mixture of modern and archaic characteristics in the Singa hominid thus reflect an African population which immediately preceded the appearance of *H. sapiens* in Africa and in the Levant (Grün & Stringer, 1991; McDermott *et al.*, 1993). In particular, its domed frontal bone shape and reduced suprorbital morphology foreshadows that of early modern humans, and identifies the Singa specimen as one of the earliest and most primitive members of the *H. sapiens* clade.

Acknowledgements

The authors wish to thank James L. Bischoff and two anonymous reviewers for providing constructive comments and suggestions which improved the manuscript. Mabs Johnston, Peter vanCalsteren and David Wright are thanked for their assistance in the NERC supported radiogenic isotope laboratory at the Open University, and Lorraine Cornish for preparing the matrix samples at The Natural History Museum. FMcD gratefully acknowledges receipt of the Bank of Ireland Newman Scholarship at University College Dublin during the course of this study.

References

Arkell, A. J., Bate, D. M. A., Wells, L. H. & Lacaille, A. D. (1951). *Fossil Mammals of Africa* Vol. 2, pp. 1–50. London: British Museum Natural History.

Bate, D. M. A. (1951). The mammals from Singa and Abu Hugar. In (A. J. Arkell, D. M. A. Bate, L. H. Wells & A. D. Lacaille, Eds) *Fossil Mammals of Africa*, Vol. 2, pp. 1–28, London: British Museum Natural History.

Bischoff, J. L. & Fitzpatrick, J. A. (1991). U-series dating of impure carbonates: an isochron technique using total sample dissolution. *Geochim. Cosmochim. Acta* **55,** 543–554.

Brothwell, D. R. (1974). The Upper Pleistocene Singa skull: a problem in palaeontological interpretation. In (W. Bernhard & A. Kandler, Eds) *Bevölkerungsbiologie*, pp. 534–545 Stuttgart: Gustav Fischer.

Clark, J. D. (1988). The Middle Stone Age of East Africa and the beginnings of regional identity. *J. World Pre-Hist* **2,** 235–305.

Goldstein, D. B., Ruiz-Linares, A., Cavalli-Sforza, L. L. & Feldman, M. W. (1995). Genetic absolute dating based on microsatellites and the origin of modern humans. *Proc. Natl. Acad. Sci. USA* **92,** 6723–6727.

Grün, R. (1986). Beta dose attenution in thin layers. *Ancient TL* **4,** 1–8.

Grün, R. & Stringer, C. B. (1991). ESR dating and the evolution of modern humans. *Archaeometry* **23,** 153–199.

Grün, R. & McDermott, F. (1994). Open system modelling for U-series and ESR dating of teeth. *Quat. Geochron.* (QSR) **13,** 121–125.

Grün, R. & Brumby, S. (1994). The assessment of errors in the past radiation doses extrapolated from ESR/TL dose response data. *Radiat. Measure.* **23,** 307–315.

Horai, S., Hayasaka, K., Kondo, R., Tsugane, K., & Takahata, N. (1995). Recent African origin of modern humans revealed by complete sequences of hominoid mitrochondrial DNSs. *Proc. Natl. Acad. Sci. USA* **92,** 532–536.

Kauffman, A. (1993). An evaluation of several methods for determining ^{230}Th/U ages in impure carbonates. *Geochim. Cosmochim. Acta* **57,** 2303–2317.

Ludwig, K. R. & Titterington, D. M. (1994). Calculation of ^{230}Th/U isochrons, ages and errors. *Geochim. Cosmochim. Acta* **58,** 5031–5042.

Luo, S. and Ku, T. L. (1991). U-series isochron dating: a generalised method employing total-sample dissolution. *Geochim. Cosmochim. Acta* **55,** 555–564.

McDermott, F., Grün, R., Stringer, C. & Hawkesworth, C. (1993). Mass-spectrometric U-series dates for Israeli Neanderthal/early modern hominid sites. *Nature* **363,** 252–254.

Nei, M. (1995). Genetic support for the out-of -Africa theory of human evolution. *Proc. Natl. Acad. Sci. U.S.A.* **92,** 6720–6725.

Rightmire, G. P. (1984). *Homo sapiens* in Sub-Sarahan Africa. In (F. Smith & F. Spencer, Eds) *The Origins of Modern Humans*, pp. 295–325. New York: Alan R. Liss.

Rosholt, J. N. (1976). ^{230}Th/^{234}U dating of travertine and caliche rinds. GSA *Abstr. Prog.* **8,** 1076.

Schwarcz, H. P. & Latham, A. G. (1989). Dirty Calcites 1. Uranium-series dating of contaminated carbonates using leachates alone. *Chem. Geol.* **80,** 35–43.

Smith, F. H. (1993). Models and realities in modern human origins: the African fossil evidence. In (M. Aitken, C. B. Stringer & P. Mellars, Eds) *The Origin of Modern Humans and the Impact of Chronometric Dating*, pp. 234–248. New Jersey: Princeton University Press.

Stringer, C. B. (1979). A re-evaluation of the fossil human calvaria from Singa, Sudan. *Bull. Br. Mus. nat. Hist.* London (Geol) **32 (1),** 77–83.

Stringer, C. B., Cornish, L. & Stuart-Macadam, P. (1985). Preparation and further study of the Singa skull from Sudan. *Bull. Br. Mus. nat Hist.* (Geol.) **38,** 347–358.

Titterington, D. M. & Halliday, A. N. (1979). On the fitting of parallel isochrons and the method of maximum likelihood. *Chem. Geol.* **26,** 183–195.

Tobias, P. V. (1968). Middle and Upper Pleistocene members of the genus *Homo sapiens* in Africa. In (G. Kurth, Ed.) *Evolution und Hominisation*, pp. 176–194 Stuttgart: Gustav Fischer.

Woodward, A. S. (1938). A fossil skull of an ancestral Bushman from the Anglo-Egyptian Sudan. *Antiquity* **12,** 193–195.

Whiteman, A. J. (1971). *The geology of the Sudan Republic*. Oxford: Oxford Clarendon Press.