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Direct ESR dating of a Pliocene hominin from Swartkrans

Two fragments of a hominin tooth (*Australopithecus robustus*) and two bovid teeth from the Hanging Remnant of the Swartkrans Formation were analysed with ESR. Research was complicated by the fact that the samples came from a curated collection and their precise provenance is unknown. The environmental dose rate was reconstructed by a series of *in situ* gamma spectrometric measurements and elemental analyses of a range of sediment samples. U-series isotopic analyses indicated that each of the teeth had a significantly different uranium uptake history, rendering the assumptive early U-uptake and linear U-uptake models ineffective. ESR and U-series data were combined to calculate open system ages, resulting in a best estimate of 1630 ± 160 ka for the Hanging Remnant. An open-system model which provides the maximum age for given U-series and ESR measurements yielded an estimate of about 2100 ka.

Two bovid teeth from Member 2, previously estimated to be between 1·0 and 2·0 Ma, yielded age estimates of between about 100 and 200 ka. No known geochemical processes are likely to explain this severe age underestimation. We conclude that these samples are of Middle to Upper Pleistocene age and their presence in Member 2 was either due to reworking or inadequate stratigraphical discrimination of these deposits.

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Introduction

The Swartkrans hominin cave site is located north-west of Johannesburg in Gauteng Province, South Africa (Broom & Schepers, 1946; Brain, 1981). It is among the most productive hominin-bearing localities in Africa. Over 200 craniodental fossils attributable to "robust" *Australopithecus* have been recovered from Member 1 alone, with a large sample also recovered from Members 2 and 3 (Grine, 1993). Around 30 hominin specimens from Swartkrans have been referred to *Homo* (Grine, 1993).

Swartkrans also documents early hominin behaviour. Stone tools from Members 1–3 total around 900 pieces classified as Developed Oldowan type (Clark, 1990, 1993). Bones apparently used by hominins as tools have also been recovered from these Members (Brain *et al.*, 1988; Brain & Shipman, 1993). Member 3 also contained 27 burnt bones which have been interpreted as evidence for the early controlled use of fire (Brain, 1993*a*).

Although research has been carried out at the locality for over half a century, the age of the hominin-bearing and archaeologically rich deposits of the Swartkrans Formation remains uncertain. This results mostly from an absence of volcanic minerals and the inapplicability of dating methods like K/Ar and $^{40}Ar/^{39}Ar$, which have provided accurate and precise age estimates for Plio-Pleistocene sites in East Africa. The situation is exacerbated by uncertainty surrounding the stratigraphy of Swartkrans. Although the formation is divided into five Members (Butzer, 1976), opinions differ substantially over the existence of discrete chronological stages of deposition (Delson, 1988; Brain, 1993*b*). In a recent review of the stratigraphy and age of Swartkrans, Brain (1993*b*: 33) concluded "it now appears that Members 2 and 3 do not differ significantly in fauna or culture from Member 1, and that *Australopithecus robustus* remains are found in all three units, whose age is assumed to be between 1·8 and 1 million years."

A variety of relative dating methods have been applied to the Swartkrans formation. Cyclic nickpoint migration and valley flank regression provided an age estimate for the first opening of the cave of around 2·6 Ma (Partridge, 1973). However, Brain (1981) noted that there is little agreement over the reliability of such methods for dating, and they must be assumed to contain large margins of error. Early estimates of faunal correlation, comparing fossil taxa with those recovered from sites in East Africa, provided age estimates of Member 1 of between 1 and 2 Ma (Vrba, 1975). In a later study, Vrba (1985) revised her estimates to between 1·6 and 1·8 Ma. The age of Member 2 was estimated to be older than 1 Ma (Vrba, 1985). Delson (1988) found little difference in the cercopithecoid faunal composition of Members 1–3 and argued for a similar age estimate for all three of around 1·65–1·9 Ma. An attempt to correlate Swartkrans with the palaeomagnetic method was unsuccessful due to a large number of unstable samples (Brock *et al.*, 1977). Relative thermoluminescence dating assuming a linear relationship of signal intensity to age, suggested an age of 1·7 Ma for Member 1, 1·5 Ma for Member 2 and 0·85 Ma for Member 3 (Vogel, 1985). These estimates were calibrated with faunal correlation ages and presented without an estimate of precision.

Blackwell (1994), using isochron ESR, was the first to apply a numerical dating method to samples from Plio-Pleistocene deposits at the site. Her study examined tooth enamel from Members 3 and 5; three samples from Member 3 provided age estimates between 360 ± 90 ka and 440 ± 80 ka (early U-uptake, EU: see below), 650 ± 150 ka and 800 ± 150 ka (linear U-uptake, LU) and 3170 ± 750 ka and 4380 ± 850 ka (recent U-uptake, RU). Two samples from Member 5 were found to be indistinguishable and dated 120 ± 10 ka (EU), 160 ± 10 ka (LU) and 210 ± 20 ka (RU) (Blackwell, 1994; dates for sample T108e1b). Blackwell (1994) noted that her results strongly implied reworking of faunal material at the site.

Conventional ESR dating methods were applied to the neighbouring hominin cave site of Sterkfontein (Schwarcz *et al.*, 1994). Whilst there was uncertainty surrounding the provenance and external dose rates used in age calculations, this study established the applicability of ESR dating to teeth from the late Pliocene and early Pleistocene. The present study builds on previous ESR dating research and presents the results of an experimental direct dating study of early hominin tooth enamel from Swartkrans.

Samples

Hominin tooth

A small piece of enamel from the M^1 of SKW 11 was selected from the curated collections of the Transvaal Museum. SKW 11 is a partial cranium, comprising a reasonably well-preserved maxilla and fragmentary crushed left side of the basicranium. It was found in 1972 within a block of breccia that dislodged from the west wall of the Hanging Remnant. This section of Member 1 contained the bulk of the early hominin remains excavated by Broom & Robinson between 1948 and 1953. SKW 11 has been assigned to *Australopithecus* (*Paranthropus*) *robustus* (Grine, 1993). The enamel piece was naturally fractured, as discovered during laboratory preparation for ESR analysis. Thus, two fragments consisting of enamel and dentine were used for this study.

Bovid teeth

Two complete bovid teeth from the Hanging Remnant (SK 2286 and SK 2262), and two bovid teeth from Member 2 (SK 6000 and SK 14122) were also analysed in this study. The samples were selected from the collection of the Transvaal Museum. The bovid samples had been attributed to particular Members, but the exact provenance is unknown.

Experimental

The basic principles of ESR dating have recently been reviewed in detail (Ikeya, 1993; Grün, 1997, 2000*a*,*b*; Rink, 1997). Conventional ESR dating methods for tooth enamel require the destruction of a specimen. This entails the separation of enamel from dentine, grinding and sieving of the enamel, and the production of about ten aliquots for generation of a dose response curve. Teeth are among the most abundant hominin remains found in Plio-Pleistocene African sites (Curnoe, 1999*a*), but their value to science means destruction for dating is usually unfeasible. Many hominin samples contain enamel fragments or complete teeth that are cracked. Cracked enamel allows an experienced curator to remove enamel pieces and re-fit them later without visible damage to the whole tooth. Grün (1995) developed a method for ESR analysis of these fragments. This technique was successfully applied to fragments of hominin tooth enamel from the site of Florisbad, South Africa (Grün *et al.*, 1996). The two enamel fragments of SKW 11 (16·1 mg and 4·8 mg) were measured in this way. Enamel and dentine were separated mechanically. Sample preparation and ESR measurements of the bovid teeth followed conventional procedures (see Grün, 1989).

Measurement of the dose value, D_E

The hominin tooth fragments were irradiated using a calibrated ^{137}Cs gamma source with cumulative doses of: 0, 92·6, 185, 370, 740, 1481, 2221, 3702, 5183 and 6664 Gy. The bovid teeth were irradiated with 0, 11·1, 22·2, 44·3, 88·6, 177, 355, 709, 1241, 2837 and 4256 Gy. The enamel fragments were measured in a sample holder similar to that described by Grün (1995), except that the holder was mounted in a Bruker ER 218 PG1 programmable goniometer for measurement. For each dose step the fragments were measured at 10° intervals over 360°. Measurements were made over four weeks.

ESR measurements were carried out at room temperature on a Bruker ECS 106 spectrometer with a 15 kG magnet and a rectangular 4102 ST cavity. The measurement conditions are routinely used for enamel samples with relatively large signals: accumulation of between 20 and 100 scans 20·48 ms time constant, 2048 bit spectrum resolution (with a total sweep time of 20·97 s), 120 G sweep width, 2 mW microwave power, 1·015 Gpp modulation amplitude, and 10·24 ms conversion factor. Signal noise was reduced by Fourier transformation and high frequency stripping (see Grün, 1998*a*); baseline corrections were made where necessary using Bruker spectrometer software.

The ESR spectra of powdered samples show a main peak with three components

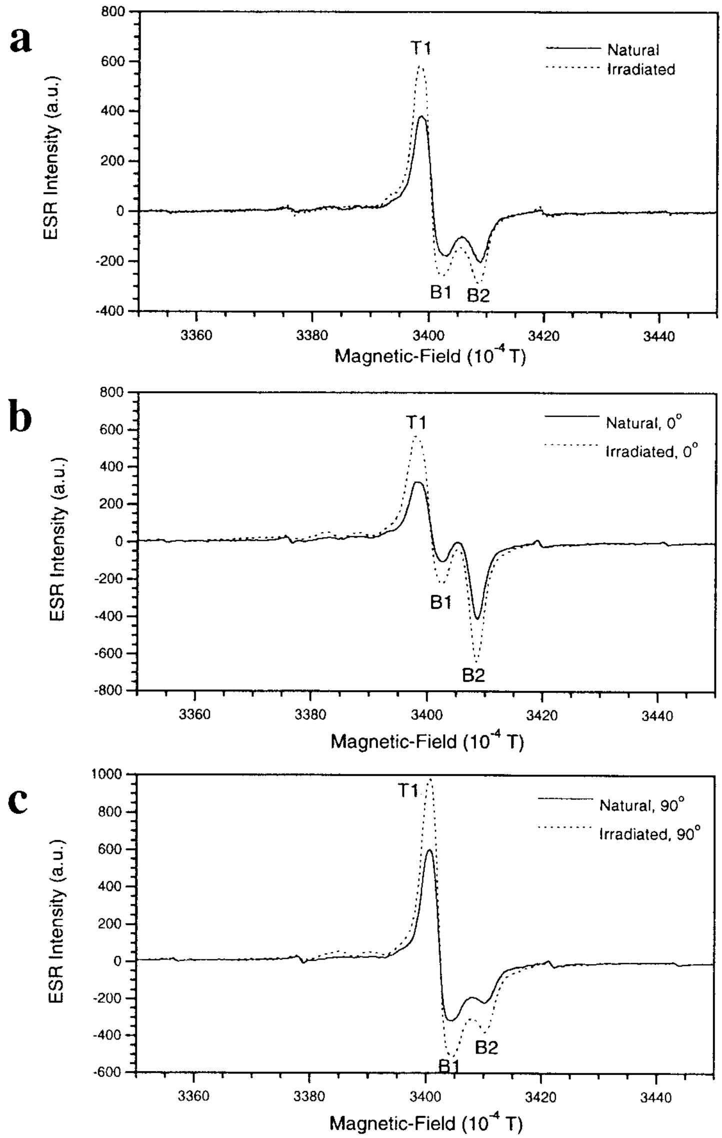

Figure 1. ESR spectra of powdered tooth enamel (a) and of fragments (b), (c).

[peak T1 and two troughs B1 and B2; see Figure 1(a)]. A single radical (CO_2^- see e.g., Vanhaelewyn *et al.*, 2000) is responsible for their generation. The relative intensity of the three components is much the same for all samples (apart from a few small interferences in the T1 and B1 regions). Recent studies of conventional methods for dose determination have suggested that the use of the first derivative of the absorption spectrum may produce erroneous results (Jonas, 1997). Methods of spectrum deconvolution have been developed to address this problem, to assess possible interference peaks in ESR spectra, and provide alternative approaches to dose calculation (Grün &

Jonas, 1996; Grün, 1998*b*). Doses (D_E-values) of the powdered bovid samples were determined in three ways (for more details, see Grün, 2000*c*):

(1) deconvolution of the integrated spectra using an ideal CO_2^- signal (Vanhaelewyn *et al.*, 2000), plus three Gaussian lines;
(2) fitting the derivative ESR spectrum with an ideal CO_2^- signal; and
(3) fitting the natural spectrum into the irradiated spectra.

We found no statistically significant differences between all three methods and the dose values presented in Table 1 are the means of the three methods (along with the quadrature of the error of the mean and the average error of the individual dose estimations).

In contrast to the powdered enamel, the ESR spectra of the tooth fragments show strong isotropic effects [Figure 1(b), (c)]. The vast majority of ESR spectra of powdered samples are similar to those shown in Figure 1(a). ESR spectra of enamel fragments may differ significantly from powdered samples at the lower end of the main peak and in the position of the first dip (B1) relative to the second dip (B2). In the enamel powder spectra, B2 is usually slightly below B1, in the single piece spectra, however, B2 may occur significantly above, below or at the same level as B1.

Tooth enamel is composed almost entirely of the orthorhombic mineral hydroxyapatite. The dominant ESR signal in hydroxyapatite (CO_2^-) has three different directional components. Two principal directions have very close magnetic properties, thus, the resulting powder spectrum approximates an axial signal (see Callens *et al.*, 1987, 1989, 1995; Vanhaelewyn *et al.*, 2000). However, crystallites in a tooth enamel fragment often have preferential orientation when an ESR signal is generated and the axial components occur with different intensities and positions in the magnetic field when the sample is rotated (see also Callens *et al.*, 1995). Because the enamel fragment does not present an ideal, single crystal, it is not possible to deconvolute the resulting ESR spectrum into its main directional components.

The doses of the fragments were estimated using the following techniques:

(1) peak-to-peak measurements (T1 to B1 and T1 to B2);
(2) deconvolution of the integrated spectrum using four Gaussian peaks; and
(3) fitting of the natural spectrum into the irradiated spectra.

All methods of estimation showed that the dose value was partly dependent upon the angle at which it was measured [see Figure 2(a) and (b)]. Fitting the natural spectrum into the irradiated spectrum had two decisive advantages over the other methods: (1) the mean dose of all angles showed the smallest standard deviation, and (2) the residual signal could be used for the assessment of the quality of the dose response curve [Figure 2(c) and (d)]. In short, the angular dose dependency can be attributed to the occurrence of interfering signals. These interferences are smallest in those samples where the residual is smallest. The dose values for SKW 11a and b were derived from those spectra where the residual was within 50% of the best fit [circles in Figure 2(a) and (b)].

Estimation of the dose rate

The gamma dose rate was measured *in situ* at six locations in the Hanging Remnant with a portable, calibrated gamma dosimeter. No sediment was attached to the teeth studied as they had been cleaned during curation. Thus, the beta dose rates were derived from the chemical analysis of sediment samples collected at the six gamma

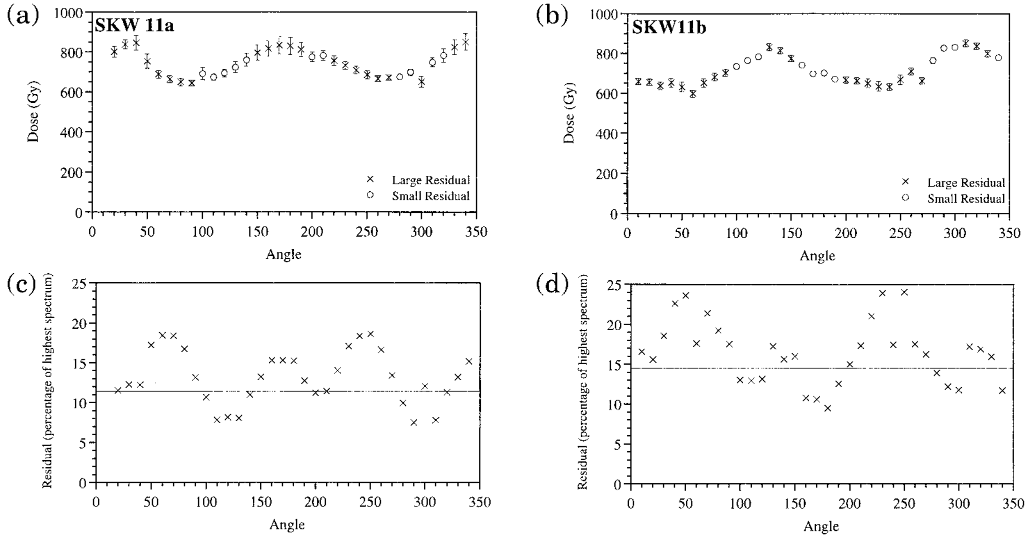

Figure 2. Dose estimations (a), (b) and residuals (c), (d) for samples SKW1a (a), (c) and SKW1b (b), (d).

dose rate locations. The results were used for the two bovid samples SK 2286 and SK 2662. The location of SKW 11 is better known and this allowed the gamma dose rate to be estimated from two *in situ* measurements in the sediment block (west wall, Hanging Remnant) from which the hominin derived. Although the vertical difference between these sampling points was about 2·5 m, the gamma dose rates are statistically indistinguishable. Two sediment samples were collected from the west wall (gamma dose rate) locations. These were analysed for the calculation of the beta dose rate (see Table 1).

The calculation of the gamma dose rate for samples SK 6000 and SK 14122 was based on one *in situ* gamma measurement. The beta dose rate was determined from analyses of a sediment sample collected at this location. The cosmic dose rates were derived from the average depths of the sedimentary units (using the attenuation data of Prescott & Hutton, 1994). Additional age calculations were carried out considering 10 ± 5 m continuous erosion of the covering sediments (after Butzer, 1976).

Isotopic concentrations in teeth (dentine only from SKW 11) and sediments were analysed with inductively coupled plasma mass spectrometry (ICP-MS) for uranium (U) and thorium (Th), and flame photometry for potassium (K). For details on ICP-MS sample preparation and measurement see Grün & Taylor (1996). Because the enamel fragments of SKW 11 could not be destroyed during the dating procedure their uranium content could not be determined directly. Therefore, a similar U-partition factor between dentine and enamel to that of the bovid teeth from the Hanging Remnant (9·4 ± 2·2) was assumed for this tooth.

Water content at all sites was found to be negligible at around 2%. Dose rate calculations utilized the conversion factors of Adamiec & Aitken (1998), alpha efficiencies of Grün & Katzenberger-Apel (1994) and Monte-Carlo beta dose attenuation factors of Brennan *et al.* (1997).

As seen below, the stand-alone ESR results (using the assumptive EU and LU models) of the three samples from the Hanging Remnant give inconclusive results.

Table 1 Results of chemical analysis, EU and LU ESR age estimates for samples from Swartkrans

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-Ḋ (µGy/a)	Sediment β-Ḋ (µGy/a)	Early U-uptake int.Ḋ (µGy/a)	Early U-uptake DE-Ḋ (µGy/a)	Early U-uptake Total Ḋ (µGy/a)	Early U-uptake AGE (ka)	Linear U-uptake int.Ḋ (µGy/a)	Linear U-uptake DE-Ḋ (µGy/a)	Linear U-uptake Total Ḋ (µGy/a)	Linear U-uptake AGE (ka)
Hanging Remnant																		
SKW11a	726 ± 49	0·64 ± 0·15*	6·02	1100	0	0·60 ± 0·03	1·21 ± 0·06	0·12 ± 0·05	262 ± 21	29 ± 5	349 ± 99	137 ± 9	777 ± 102	934 ± 138	173 ± 47	68 ± 4	532 ± 52	1365 ± 162
SKW11b	746 ± 70	0·64 ± 0·15*	6·02	1100	0	0·60 ± 0·03	1·21 ± 0·06	0·12 ± 0·05	262 ± 21	29 ± 5	349 ± 97	137 ± 9	777 ± 101	960 ± 153	172 ± 47	68 ± 4	531 ± 52	1405 ± 191
SK2286a	1179 ± 76	0·57	5·30	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	276 ± 36	98 ± 13	772 ± 103	1527 ± 226	137 ± 19	49 ± 6	584 ± 98	2019 ± 363
SK2286b	1202 ± 64	0·53	6·05	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	257 ± 33	112 ± 16	767 ± 103	1563 ± 226	128 ± 16	56 ± 8	582 ± 98	2065 ± 365
SK2286c	1088 ± 67	0·74	7·84	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	360 ± 47	145 ± 20	903 ± 109	1205 ± 163	178 ± 23	72 ± 9	648 ± 99	1679 ± 277
SK2662a	651 ± 43	0·49	5·10	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	246 ± 32	96 ± 15	740 ± 102	880 ± 134	120 ± 17	47 ± 7	565 ± 98	1152 ± 213
SK2662b	685 ± 62	0·50	4·80	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	242 ± 31	88 ± 12	728 ± 102	941 ± 157	118 ± 15	43 ± 5	559 ± 97	1225 ± 239
SK2662c	880 ± 85	1·08	5·70	1000	100	0·63 ± 0·17	2·2 ± 0·81	0·4 ± 0·4	343 ± 88	55 ± 38	533 ± 64	106 ± 14	1037 ± 116	849 ± 125	261 ± 31	52 ± 7	711 ± 101	1238 ± 213
Member 2																		
SK6000a	74·6 ± 6·3	1·34	51	900	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	42 ± 8	301 ± 43	566 ± 75	1196 ± 89	62 ± 7	151 ± 22	284 ± 38	764 ± 49	98 ± 10
SK6000b	86·6 ± 11·7	2·81	31	900	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	42 ± 8	643 ± 86	349 ± 47	1321 ± 101	66 ± 10	325 ± 45	176 ± 24	830 ± 56	104 ± 16
SK6000c	75·2 ± 6·4	0·73	61	900	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	42 ± 8	166 ± 24	681 ± 27	1176 ± 93	64 ± 7	83 ± 13	342 ± 45	754 ± 52	100 ± 11
SK14122a	91·1 ± 2·5	1·40	23	1000	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	38 ± 7	372 ± 50	262 ± 34	959 ± 64	95 ± 7	183 ± 26	130 ± 18	638 ± 38	143 ± 9
SK14122b	94·9 ± 5·7	1·17	23	1000	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	38 ± 7	322 ± 45	269 ± 45	916 ± 60	104 ± 9	158 ± 23	132 ± 17	615 ± 36	154 ± 13
SK14122c	119·8 ± 2·8	0·73	15	1000	100	0·30 ± 0·02	2·3 ± 0·12	0·27 ± 0·02	287 ± 21	38 ± 7	235 ± 34	198 ± 27	758 ± 49	158 ± 11	113 ± 17	96 ± 13	534 ± 31	224 ± 14

For details on ESR age calculation and U-uptake modes see Grün (1989). EN=enamel; DE=dentine; *internal dose rate calculated from dentine U-concentration and dentine/enamel U-partition factors of samples SK2286 and SK2662 (9·4 ± 2·2). TT=total enamel thickness; S1/S2=surface layer removed from each side of the enamel samples. Error in D_E after Grün & Brumby (1994); alpha efficiency: 0·13 ± 0·02 (Grün & Katzenberger-Apel, 1994), beta attenuation from Monte Carlo data of Brennan *et al.* (1997); initial $^{234}U/^{238}U$ from Table 2; sediment results from measurement of four representative samples. Gamma dose rate from six *in situ* measurements in the Hanging Remnant. Cosmic dose rate assumes 10 ± 5 m continuous erosion of covering sediments (using data from Prescott & Hutton, 1994).

To investigate this further, a small sample of dentine from SKW 11 (<1 mg), and enamel and dentine from the bovid teeth from the Hanging Remnant, were analysed for U-series isotopes. For details of U-series isotope analysis see Grün *et al.* (1999). This allows for the U-uptake history to be modelled (e.g., Grün & McDermott, 1994; Grün *et al.*, 1999).

Results and discussion

Hominin tooth

Figure 2 shows the dose estimates at the various angles using fitting of the natural spectrum into the irradiated spectra. The average dose of all estimates of the two fragments is: SKW 11a 742 ± 72 Gy and SKW 11b 716 ± 89 Gy (the error being the quadrature of the average error of the individual dose estimation and the standard deviation of the mean dose of all angles). The lowest residuals for sample SKW 11a are in the lower dose region. For SKW 11b the lowest residuals are found in the higher dose region. Applying the 50% threshold [see Figure 2(c) and (d)] the resulting dose values are: SKW 11a 726 ± 49 Gy and SKW 11b 746 ± 70 Gy.

Age of the Hanging Remnant

As stated previously, without knowledge of the actual U-uptake history of a tooth the most likely ESR age usually lies between the EU and LU estimates (e.g., reviews by Grün, 1989, 1997, 2000*a*,*b*). This would mean that the apparent age of the Hanging Remnant lies between about 1020 ka (weighted mean of EU age estimates) and 1400 ka (weighted mean of LU age estimates—see Table 3). This is significantly younger than has been suggested from faunal correlation (summarized above).

The following hypotheses may explain the apparently underestimated age of samples from the Hanging Remnant:

(1) ESR dating does not work at the site;
(2) the EU and LU models are inappropriate, with differences between the samples caused by differences in U-uptake history;
(3) the difference between the different age estimates is caused by the unknown provenance of the specimens and thus inadequate reconstruction of dose rates (in this study);
(4) reworking processes have led to the mixing of samples of different age; and
(5) thermal instability of the ESR centre causes significant age underestimation.

The dose values determined for sub-samples of most teeth analysed in this study agree within error. Where they do not agree, however, variation in the dose value is highly correlated with variation in the concentration of uranium for each sub-sample. For example, the higher dose in sub-sample SK 2662c can be attributed to the higher U-concentration in the enamel of this sample (compared to the two other sub-samples of this specimen). It is concluded that ESR does not produce random results for the teeth analysed from this site.

Although the errors associated with the estimated external dose rates are large, they cannot account for differences in the LU and EU age estimates for the two bovid teeth (SK 2286 and SK 2262). To address this problem a small amount of enamel and dentine was analysed for U-series isotopes. This allowed for the calculation of closed system U-series ages, as well as the calculation of combined, open system U-series/ESR (US/ESR) ages using the *P*-value system of Grün *et al.* (1988) and the CS-US/ESR model of Grün (2000*d*). The parametric *P*-value system uses the ESR and U-series results to model a continuous U-uptake function. In contrast, the CS-US/ESR model assumes that the uranium measured today migrated into the specimen at a time that corresponds to the closed

Table 2 Results of U-series analysis (activity ratios; 1-σ errors)

Sample no.	U (ppm)	$^{234}U/^{238}U$	$^{230}Th/^{232}Th$	$^{230}Th/^{234}U$	Age (ka)
SKW11 DE	9·95	1·926 ± 0·113	190 ± 21	0·600 ± 0·066	91^{+33}_{-26}
SK2286B EN	0·609	1·392 ± 0·068	220 ± 7	1·048 ± 0·031	342^{+73}_{-46}
SK2286C EN	0·843	1·480 ± 0·072	306 ± 25	1·087 ± 0·085	391^{+433}_{-125}
SK2286C DE	6·65	1·442 ± 0·002	548 ± 7	1·013 ± 0·011	284^{+12}_{-11}
SK2262A EN	0·593	1·300 ± 0·111	275 ± 4	0·9066 ± 0·0141	212^{+22}_{-18}
SK2262B EN	0·621	1·217 ± 0·039	54 ± 1	0·9151 ± 0·0231	226^{+21}_{-18}
SK2262A DE	5·96	1·108 ± 0·001	478 ± 18	0·9321 ± 0·0263	258^{+32}_{-25}

Isotopic values were converted to activity ratios using the following decay constants: $\lambda_{230}=9{\cdot}195\ 10^{-6} \pm 7{\cdot}19\ 10^{-8}\ a^{-1}$; $\lambda_{234}=2{\cdot}835\ 10^{-6} \pm 5{\cdot}71\ 10^{-9}\ a^{-1}$; $\lambda_{238}=1{\cdot}551\ 10^{-10} \pm 1{\cdot}66\ 10^{-13}\ a^{-1}$.

system U-series age. The CS-US/ESR age is the *maximum possible age* that can be calculated from the combination of ESR and U-series values. There are specific geochemical conditions where this model seems applicable (e.g., Grün *et al.*, 2001).

Table 2 shows U-series results for the samples from the Hanging Remnant. The errors associated with thermal ionization mass spectrometry (TIMS) U-series analyses are usually very small. The small amount of enamel and dentine available for analysis resulted in relatively high errors for results reported here. It is evident that the U-series results for each sample are very different: the apparent closed system age of the dentine of SKW 11 is in the range of 90 ka; the ages of all components of SK 2286 are >280 ka; and ages of SK 2262 are between 210 and 260 ka. The age results on the dentine and enamel for each tooth are similar. The differences in the U-series ratios between the teeth have significant bearing on open system age calculations.

It should be noted that the computer programmes normally used to calculate uranium uptake in ESR dating were stretched beyond their practical limits when applied to U-series analyses for samples from Swartkrans. This is because the mathematical procedures in the computer programme used do not allow for the calculation of open system ages for $^{230}Th/^{234}U$ ratios of >1 (sample SK 2286) and *P*-values of >8 (SKW 11). To address this problem, SKW 11 values were run for dose values that approached the limits of the model. At high *P*-values the US/ESR age calculations approximate the CS-US/ESR model ages (see also Grün & Schwarcz, 2000). For SKW 11 the US/ESR age is 8% smaller than the CS-US/ESR age for *P*=7·5. A similar age difference was assumed for the final age estimates of sample SKW 11. For sample 2286, a $^{230}Th/^{234}U$ ratio of 0·99 was used with $^{234}U/^{238}U$ ratios for enamel and dentine that result in the same closed system U-series ages as the measured ratios.

The calculations show that for SK 2286 the EU model is closer to the open system (US/ESR) model age, but for SK 2262 the LU model is more appropriate (Table 3). The US/ESR results of these samples yield much better agreement between age calculations than the simple assumptive models of EU and LU. The hominin tooth, however, is characterized by a U-uptake history that is different from those determined for the bovid teeth (as documented by the small $^{230}Th/^{234}U$ ratio in the dentine of 0·600 ± 0·066). The US/ESR and CS-US/ESR age calculations give similar results of around 2 Ma.

Because of their unknown provenance, it is impossible to know whether the differences in age between the Hanging Remnant samples is due to reworking or inadequacies in the external dose rate calculations. If

Table 3 Summary of open system calculations (in ka)

	EU	LU	US/ESR	CS-US/ESR
SKW 11	950 ± 150	1390 ± 180	~2020 (P>13)*	2200 ± 260
SK2286	1430 ± 210	1920 ± 340	1420 ± 250†	2580 ± 650
SK2262	890 ± 140	1210 ± 220	1190 ± 400	1650 ± 440
Weighted mean	1020 ± 90	1400 ± 130	1630 ± 160	2110 ± 210

*Estimated from approximations to $P \sim 8$.
†Calculated for $^{230}Th/^{234}U = 0{\cdot}99$ and $^{234}U/^{238}U$ ratios corresponding to the closed system U-series ages of Table 2.

reworking took place it was at a relatively early stage in the sedimentation history of this unit. Given that the most likely age (as determined from the combined U-series/ESR model) are close to faunal estimates for the Hanging Remnant, there is no reason to assume that thermal instability has led to a systematic age underestimation. To put it another way, the reasonable agreement of US/ESR results and age estimates from other (independent) methods suggest that thermal stability of the ESR centres is far in excess of the average US/ESR age of 1·6 Ma.

To summarize these results in light of the above working hypotheses, for samples from the Hanging Remnant:

(1) ESR dating does work;
(2) significant differences in the U-uptake histories (documented by measured U-series isotopes) explain some of the larger differences between the (assumptive) EU and LU age estimates;
(3) the differences in the US/ESR ages are most likely due to the assumptions in the external dose rate calculations or to reworking (although, if the latter has occurred, it was early in the sedimentation history of the Hanging Remnant);
(4) an age underestimation due to thermal fading, should it have occurred, is on a small scale; and
(5) the best age estimate is provided by the US/ESR age model (1630 ± 160 ka) but the Hanging Remnant is younger than the maximum CS-US/ESR calculation (2110 ± 210 ka).

The correct age of these teeth may be older than suggested here as all age calculations are based on present day (negligible) water contents of tooth enamel, dentine and sediment samples. Butzer (1976, 1984) suggested Swartkrans has a complicated hydrological history that includes periodic exposure of deposits to high intensity rains of considerable duration. It is not possible to translate these observations numerically for age calculations in this study. Therefore, the age of the samples may be somewhat older (in the range of 10%) than the US/ESR dating results presented here.

At least 10 fossils from the Hanging Remnant have been attributed to *Homo* (Clarke *et al.*, 1970; Brain *et al.*, 1974; Clarke, 1977; Olson, 1978; Susman, 1988; Grine & Strait, 1994). The age found here for this unit is broadly contemporaneous with the suggested age of *H. habilis* (Stw 53) from Skerkfontein (Vrba, 1975; Stiles & Partridge, 1979; Butzer, 1984; Tobias, 1988; Clarke, 1994; Kuman, 1994; Curnoe, 1999*b*).

Member 2

The EU and LU ESR ages of the two bovid teeth from Member 2 are between about

100 and 200 ka. This is significantly lower than the postulated age of this unit of between 1 and 2 Ma (summarized above). If observations made in this study for samples from the Hanging Remnant are applied to those of Member 2, we find:

(1) the ESR ages are neither too young because ESR does not work nor because the thermal stability of the ESR signal leads to systematic age underestimation;
(2) differences in U-uptake histories may explain the differences between the EU and LU model ages of different teeth (the samples with the lower U dose rates yield older LU age estimates: a sign that the U-uptake was delayed more than provided by LU, see Grün, 2000*d*);
(3) even if the extremely unlikely RU uptake model [U-uptake is such a recent process that the U-dose rates in enamel and from dentine are negligible (Blackwell, 1994)] yields only ages of <370 ka;
(4) none of the sediment analyses indicate that extremely low environmental dose rates (resulting in an average total dose rate of between 40 and 80 μGy/a) were prevalent for most of the burial life of the samples; thus
(5) the only conclusion that can reasonably be made is that the samples are significantly younger than 1 Ma.

It is not possible to assess whether these teeth were reworked from younger deposits (e.g., Member 5) or whether the sediments from which the samples were recovered are actually significantly younger than previously assumed.

Conclusions

The age estimates reported in this study may be improved significantly with ESR and U-analyses of more samples, particularly those collected *in situ*. The best age estimate for the Hanging Remnant is given here by the US/ESR age estimate of 1630 ± 160 ka, the maximum possible age being 2110 ± 210 ka. Together with results for Sterkfontein (Schwarcz *et al.*, 1994; Curnoe, 1999*b*), it is concluded that ESR can provide reasonable age results for samples of late Pliocene/early Pleistocene age, at least for sites where the environmental dose rate is reasonably low.

The age of the two bovid teeth from Member 2 of the Swartkrans Formation is significantly lower than previously estimated. No known geochemical processes may account for this difference. Whether it is due to reworking or the fact that the sediments from which the samples were recovered are significantly younger than 1 Ma cannot be determined presently.

This ESR dating study shows again that the assumptive EU and LU ESR age models are too simplistic. With the application of commonly accessible (TIMS) U-series analyses combined US/ESR age estimates should be calculated for important palaeontological/archaeological sites. Given the fact the exact sample locations of faunal remains are often poorly recorded, it seems timely to further develop non-destructive dating methods. Because ESR (particularly in combination with U-series dating) can provide reliable age estimates (see also Thorne *et al.*, 1999), it is not extravagant to suggest that hominin samples of a few 100 mg be sacrificed for more precise dating. The accurate timing of human evolution is, in the end, dependent upon the direct dating of human remains.

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