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ESR dating of the Die Kelders Cave 1 Site, South Africa

ESR measurements were made on ten enamel subsamples from six teeth recovered in layers 4–5, 6, 10, and 12 in the site of Die Kelders Cave 1, South Africa. The teeth (enamel and dentine) contained significant concentrations of uranium and therefore the U uptake model has a large influence on the computed ages. Variations in moisture content in the sediment had a smaller effect on the dose rate and calculated ages. For any given model of U uptake and moisture content, all the teeth gave very similar ages, implying that the entire deposit was formed over a short interval (<10,000 y). Comparison with OSL ages for the sediments suggests that the teeth experienced early U uptake, in which case the average age of the deposit is 70 ± 4 ka (assuming a moisture content of 10%). Agreement between replicate subsamples was excellent.

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Introduction

The site of Die Kelders is located on the southern coast of South Africa, in the Western Cape Province, about 120 km south of Cape Town, and about 2 km N of the fishing village of Gansbaai. The Die Kelders site is in one of a network of caves of partly karstic origin, partly enlarged by the sea. The site currently under investigation is in the cave identified as DK1. The archaeological deposits form a series of sedimentary layers partly filling this large sea-facing opening. A complete description of the stratigraphy of the site is given by Marean *et al.* (2000), following on earlier descriptions by the first excavators at the site, Tankard & Schweitzer (1974, 1976).

The cave occurs near the contact between the underlying Paleozoic quartzites of the Table Mountain Sandstone, and the Cenozoic Bredasdorp Group. The latter consists largely of sandy limestones and calcite-cemented sandstones. The sedimentary fill of the cave is capped by a shell midden 1·5 m thick containing LSA (Late Stone Age) artefacts as well as pottery and debris from sheep tending. Layers 2–5 (following the original designation of Tankard & Schweitzer, 1974) consist of shelly and sterile sand. Layers 6 to 13 contain a MSA (Middle Stone Age) industry and vertebrate fossils including some hominid remains. These layers are dominantly composed of weakly cemented or uncemented sand colored in varying tones of yellow, brown, gray and white, and containing varying amounts of rock-fall blocks which have largely leached and decomposed into masses of variegated sand giving a "fruitcake" appearance to the enclosing layers. The bones in these layers are also partly decomposed, but relatively fresh teeth have been obtained in the course of excavation.

Previous dating studies of these deposits (Avery *et al.*, 1997) showed that they were deposited between 60 and 80 ka, beyond the range of radiocarbon dating. Various methods now exist to determine the age of archaeological sites in this time range, based on measurement of the growth of

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radiation-induced signals in natural materials that had been deposited at a site in a signal-free ("zeroed") state. In this paper we will discuss the application of one of these methods, electron spin resonance (ESR) dating of tooth enamel, while Feathers & Bush (2000) have used optically stimulated luminescence (OSL) and thermoluminescence (TL) dating of quartz and feldspar from the sediment.

Tooth enamel, which consists of the mineral hydroxyapatite, displays a radiation-induced signal with a g-value of 2·0018, with a lifetime greater than 10^9 years (Schwarcz, 1985), that can be used to determine the age of mammalian teeth at archaeological sites (Rink, 1997). We previously have used this method to obtain a preliminary estimate for the age of part of the MSA section at Die Kelders (Avery *et al.*, 1997). In this paper we extend these results to other parts of the MSA section.

ESR dating

The ESR dating method is based on the assumption that the intensity of the ESR signal in enamel grows monotonically during the burial history of the tooth, as a result of bombardment by environmental radiation from U, Th, and K in the tooth and sediment as well as cosmic rays. The apparent dose that the tooth has received since burial, called the equivalent dose (D_E), is determined by the method of additive dose. We measure the intensity of the ESR signal that was present in the sample at the time of its collection and then observe the growth of this intensity as a function of added laboratory doses of gamma radiation. The equivalent dose is determined by back-extrapolating the intensity curve to zero intensity; the intersection on the dose-axis represents the equivalent dose that the enamel had already received at the time of collection.

If the external and internal dose rates were constant then the age would be given by the ratio of D_E (in Gy) to the total dose-rate (in Gy/a). More generally, the age is computed by the solution of an integral equation in which the dose-rate is allowed to change through time. This change occurs mainly because part of the dose is produced by U atoms inside the teeth which have been chemically absorbed from the adjacent sediment and ground-water during the burial history. For teeth whose self dose is a significant fraction of their total dose, the dose rate may increase through time for two reasons: (a) as a result of time-dependent U-uptake; and (b) as a result of growth of daughter radioisotopes produced by the *in situ* decay of U atoms. In general we assume that the U uptake history lies between two limiting models: early uptake (EU) in which all of the U is taken up by enamel and dentine soon after deposition of the teeth; and linear uptake (LU) in which the U content of the tooth is assumed to have increased at a constant rate since its burial (Grün *et al.*, 1987). In most instances the self dose is dominated by beta rays derived from the adjacent dentine, while another beta dose consists of external beta rays from adjacent sediment in direct contact with enamel. This beta dose is attenuated as it passes through the enamel, in a fashion which can be modeled by beta-ray transport theory. In this paper we have used a newly developed method to calculate this attenuation called One-Group Theory, which is incorporated into a new computer software called ROSY (Brennan *et al.*, 1997*b*). External cementum layers, which might also attenuate sediment-derived beta rays, were not present on the teeth analyzed here.

Methods

Samples of teeth were recovered from the site in the course of excavations during 1993 and 1995. Sediment found immediately adjacent to each tooth was also collected for measurement of its radioactivity. Enamel and adjacent dentine were isolated from the teeth. A layer a few micrometers thick was stripped from each sample of enamel to remove the influence of alpha particles from adjacent sediment. The concentrations of uranium (U) in the enamel and dentine were determined by delayed neutron activation analysis in the McMaster Nuclear Reactor.

Thermoluminescence dosimeters were emplaced at some nearby sites but it has not yet been possible to obtain dose rates from these. We have therefore used neutron-activation analyses of U, Th, and K in the adjacent sediment to construct the gamma ray dose from a sphere of sediment 30 cm in radius, surrounding each sample. The sediment at this site is relatively homogeneous sand of uniform grain size; it would be characterized as a "smooth" rather than "lumpy" site in the sense of Schwarcz (1994) and Brennan *et al.* (1997a), implying that the dose rate as calculated from the composition of the sediment is likely to be representative of the true gamma dose experienced by the sample. The sediment-derived dose of beta rays was also computed from the U, Th, K analyses of attached sediment. In view of the homogeneous character of this site moisture contained in the sediment can significantly reduce the dose-rate of gamma and beta rays. The sediment at this site consists of sand which is very porous and permeable; it is therefore difficult to estimate what has been the long-term water content of the sediment. We have used three trial values of 10, 20 and 30 weight % water which spans the maximum possible range experienced by the teeth. The cosmic ray dose rate, which can range up to several percent of the total dose rate, depends on the depth of burial of the tooth, but also on the extent of cover by the overhanging bedrock at the cave. The samples collected in 1993 and 1995 all came from sites well under the cosmic ray "shadow" of the overhanging cliff and therefore were almost completely shielded from cosmic rays. Therefore the external dose was dominated by gamma radiation from the sediment.

Samples

The site locations and stratigraphic assignments of the sample are listed in Table 1. They are teeth of unspecified bovids (eland or other smaller species) derived from layers 4–5, 6, 10 and 12, and consist of fragments of whole teeth including attached dentine, but lacking any cementum. They are all fully erupted teeth showing some degree of wear. In addition, we have recalculated ages for the two conjoining fragments of a single eland tooth discussed in Avery *et al.* (1997), which were found in two different stratigraphic units: level 4–5 and level 6. Two separate subsamples of enamel were analyzed from three of the teeth as a further check on the consistency of the data.

Results

The analytical data on which the age calculations are based are presented in Table 1. The dose rate calculations and ages are presented in Table 2 and summarized in Figure 1. For each sample or subsample, we give EU and LU dates for assumed moisture content (M%) values of 10, 20, and 30%, to demonstrate the effect on the ages of varying moisture. For the minimum moisture content of 10%, the ages range between 64 and 78 ka with the exception of one outlier (95391) from layer 10 which gave an EU age of 99 ka. The EU ages give the minimum ages for the EU/LU set of assumptions (Rink, 1997). The range of the other nine samples (for the same assumptions of U uptake and moisture content) is surprisingly small given that they span most of the stratigraphic range of the archaeological record at this site (from layers 4 to 12). This suggests that these deposits were formed in a relatively short time. There is no tendency

Table 1 Die Kelders tooth samples

Sample	DK# (ESR-field#)	Strat. unit	U en* ppm	U den* ppm	U sed* ppm	Th sed† ppm	K sed† %	Thickn.‡ µm	Rem (1)§ µm	Rem (2)‖ µm	Cosmic burial¶ m
95038-1A	5-5325	4/5	0·24 ± 0·02	34·01 ± 27·10*	2·55 ± 0·25	5·46 ± 0·13	0·25 ± 0·01	1168 ± 121	58 ± 29	60 ± 30	24
95038-2A		4/5	0·21 ± 0·10	27·10 ± 0·10				1181 ± 161	44 ± 22	40 ± 20	
95038-2B	5-5325	4/5	0·29 ± 0·10	29·57 ± 0·10	2·55 ± 0·24	5·46 ± 0·13	0·25 ± 0·01	996	39	48	24
								169	19	24	
95389	21-5880	4/5	0·40 ± 0·10	29·02 ± 0·10	1·82 ± 0·10	3·54 ± 0·15	0·13 ± 0·01	902 ± 89	60 ± 30	59 ± 30	24
95037A	6-5363	6	0·17	33·57	3·37 ± 0·34	4·52 ± 0·11	0·26 ± 0·01	1331	74 ± 37	37 ± 19	24
95037B		6	0·17	34·29 ± 0·10				967	27 ± 16	40 ± 20	
95391	43-9089	10	0·44 ± 0·10	38·86 ± 0·10	1·73 ± 0·10	2·37 ± 0·11	0·10 ± 0·01	1143 ± 66	32 ± 16	48 ± 24	24
95390	51-9181	12	0·30 ± 0·10	40·97 ± 0·10	5·62 ± 0·10	8·79 ± 0·23	0·16 ± 0·01	1075 ± 62	51 ± 25	34 ± 17	24
95392A	45-9055	12	0·24 ± 0·10	43·12 ± 0·10	4·95 ± 0·10	6·61 ± 0·20	0·16 ± 0·05	922 ± 112	25 ± 12	77 ± 38	24
95392B		12	0·24 ± 0·10	45·01 ± 0·10				1091 ± 76	67 ± 33	62 ± 31	

*U en, U den, U sed = uranium concentration in enamel, dentine and sediment, (ppm) = parts per million.
†Th sed, K sed = thorium concentration in sediment, potassium concentration in sediment, (%) = (weight %).
‡Thickn. = thickness of enamel.
§Rem (1) = thickness of enamel stripped from side in contact with sediment.
‖Rem (2) = thickness of enamel stripped from side in contact with dentine.
¶Burial = estimated overburden thickness in metres used for calculating the cosmic dose rate.

Table 2 Die Kelders: ESR dates

			EU				LU			
Sample	D_E Gy	Water %	alpha	beta	gamma +cosmic	Age ka	alpha	beta	gamma +cosmic	Age ka
95038-1A	74·97 ± 2·18	10	52	478	574	67·9 ± 5·1	24	273	574	86·0 ± 7·2
		20	54	474	521	71·5 ± 5·2	25	268	521	92·2 ± 7·2
		30	55	471	477	74·8 ± 5·2	25	264	477	97·9 ± 7·2
95038-2A	75·97 ± 2·22	10	48	407	574	73·9 ± 6·0	22	239	574	91·0 ± 8·1
		20	49	402	521	78·1 ± 6·2	22	233	521	97·9 ± 8·2
		30	50	399	477	82·1 ± 6·2	23	228	477	104·3 ± 8·2
95038-2B	80·36 ± 2·03	10	64	501	574	70·5 ± 5·9	30	291	574	89·8 ± 8·2
		20	66	496	521	74·2 ± 6·1	30	285	521	96·1 ± 8·4
		30	67	492	477	77·5 ± 6·2	31	279	477	102·1 ± 8·4
95389	72·2 ± 0·6	10	92	495	371	75·4 ± 4·3	44	276	371	104·5 ± 6·3
95389	72·2 ± 0·6	20	93	492	337	78·3 ± 4·5	45	272	337	110·4 ± 6·6
		30	95	489	306	81·1 ± 4·6	46	269	306	116·3 ± 6·9
95037A	74·71 ± 1·76	10	38	423	616	69·4 ± 5·9	17	249	616	84·7 ± 8·2
		20	39	418	559	76·7 ± 6·1	18	243	559	91·2 ± 8·3
		30	39	415	511	77·4 ± 6·1	18	238	511	97·4 ± 8·3
95037B	83·87 ± 2·45	10	37	586	616	67·7 ± 5·7	17	341	581	86·1 ± 8·2
		20	38	580	559	71·3 ± 5·9	18	333	559	92·2 ± 8·3
		30	39	575	511	74·5 ± 5·9	18	327	511	97·9 ± 8·3
95391	100·0 ± 1·1	10	114	578	314	99·4 ± 4·7	56	310	314	147·1 ± 7·6
		20	116	576	285	102·3 ± 4·9	57	308	285	153·9 ± 7·9
95391	100·0 ± 1·1	30	117	575	261	104·9 ± 5·0	58	306	261	160·0 ± 8·0
95390	132·0 ± 4·5	10	68	694	1025	73·8 ± 5·0	30	426	1025	89·1 ± 7·3
		20	70	684	930	78·4 ± 5·2	32	413	930	96·0 ± 7·3
		30	72	676	851	82·6 ± 5·3	32	402	851	102·7 ± 7·6
95392A	121·0 ± 5·6	10	53	886	855	67·5 ± 5·1	24	556	855	84·4 ± 6·9
95392a	121·0 ± 5·6	20	54	870	776	71·2 ± 5·4	25	536	776	90·5 ± 7·3
		30	55	856	710	74·6 ± 5·6	25	521	710	96·3 ± 7·7
95392B	106·0 ± 1·5	10	51	763	855	63·5 ± 3·8	23	487	855	77·7 ± 5·3
		20	52	747	776	67·3 ± 4·0	24	468	776	83·6 ± 5·6
		30	53	735	710	70·7 ± 4·1	24	454	710	89·2 ± 5·8

*D_E = equivalent dose.

†M% = (weight of moisture/weight of dry sediment) × 100.

‡alpha, beta, gamma + cosmic = alpha, beta, gamma + cosmic dose rates in enamel (10^{-6} Gy/year).

§The beta and gamma dose rates to enamel were calculated from sediment attached to teeth using the conversion data of Nambi & Aitken (1986) and the moisture correction factors of Aitken (1985).

||The beta dose rate to enamel was calculated using the ROSY algorithm based on One-Group theory (O'Brien *et al.*, 1964).

¶The cosmic dose rates to enamel based on overburden thickness were calculated using the data of Prescott & Hutton (1988).

for the ages to increase with stratigraphic depth (indeed, there is a small but insignificant trend to decreasing age with depth). There is excellent agreement between subsamples of the same teeth. The single outlier (95391) displayed an equivalent dose that was similar to that of the other samples, but was buried in sediment with significantly lower content of radioactive elements (especially U and Th) which leads to a higher calculated age. Also, samples 95037 and 95038, although found in different strata (4–5 and 6, respectively) were found to be conjoining fragments of the same tooth. The two samples gave identical ages (EU and LU). The significance of this observation is unclear. Presumably the tooth was deposited in a fragmentary state in one

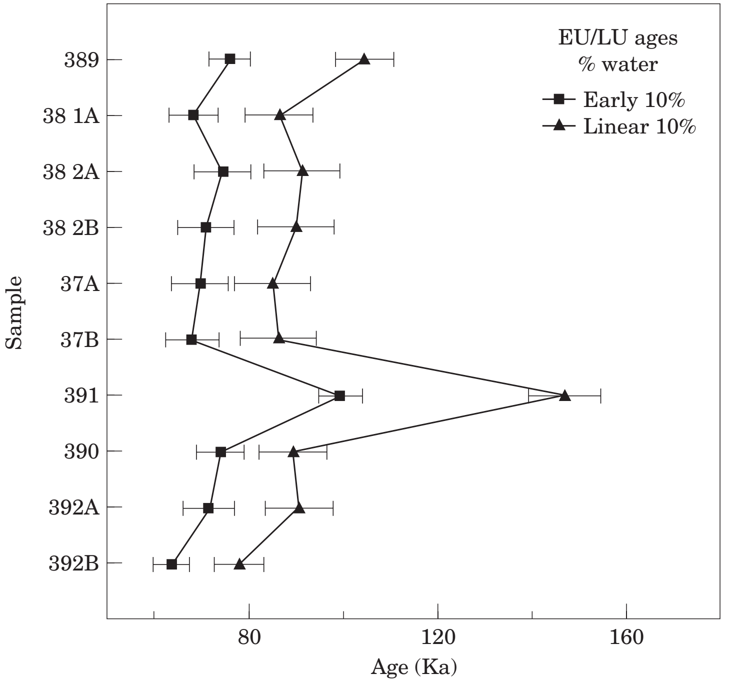

Figure 1. Distribution of ages with depth in Die Kelders site.

of these layers and one of the fragments migrated either upwards or downwards in the stratigraphic sequence. Since the environmental and internal dose rates of the two samples are similar, it is not possible to tell which sample migrated away from its original site or when. The data are consistent with the general observation that the sediments at this site were deposited rather quickly.

The age of deposition indicated by the ESR measurements depends on the selection of the appropriate model parameters to describe the dose-rate history of the samples: U uptake and moisture content (M%). In respect to U uptake, we note first of all that a significant fraction of the total dose received by the teeth came from U in the enamel and dentine. As a result, there is a rather large difference between the EU and LU ages for these teeth. Furthermore, we must note that linear uptake is only one possible model to describe the U uptake: the actual uptake history may be transitional between EU and LU. It is possible to define this U uptake history more precisely by uranium-series analysis (^{230}Th/^{234}U, ^{234}U/^{238}U ratios) of the enamel and dentine (Grün *et al.*, 1988), but we have not yet made such analyses of these teeth. We can, however, consider the distribution of the ages themselves and make some inferences about the effects of possible values for model parameters.

Moisture content: (M%)

We see from Figure 1 and Table 2 that, over the 10–30% range used here, the age increases by an average of about 10% with moisture content. The present day moisture content is a few percent, but is clearly not representative of the conditions prevailing during most of the burial history. Feathers & Bush (2000) have argued that, assuming that the sediment was a sand-loam mixture, the water content at saturation would be no more than 12%. However, this is likely to have varied depending on the texture, and could have been larger in more porous parts of the sediment. Moreover, M% is likely to have varied through the history of the deposit, depending on the climate, (especially average annual rainfall). Lacking an independent means of estimating M%, we can only propose that the EU and LU model ages of the teeth lie somewhere within the values given for this range of moisture content. We have assumed a maximum possible water content of 30% in calculating ages for Table 2 but, as argued by Feathers & Bush (2000), a value of 10% is most likely. Higher water contents are unlikely to have persisted in the vadose zone in such permeable materials. Variation in ages shown in Table 2 for varying M% is much greater than between subsamples of the same tooth, or for that matter between almost all the samples from the site, when calculated for a single M%. Clearly, knowledge of the true average value of this parameter is crucial in establishing the age. It is interesting to note that only the choice of a low M% value (ca. 10%) brings the ESR and luminescence dates into concordance.

Uranium uptake

This is also a difficult model condition to estimate, and has a large influence on our conclusions regarding the age. In the absence of any independent data such as U-series analyses, we may get some idea of the true uptake behavior by comparing the range of ages given by either of the two models (keeping M% constant). Excluding the outlier, the average EU, 10% age is 70·1 ± 3·8 ka (cv=coefficient of variation=5·5%) while the LU, 10% age is 88·1 ± 7·3 (cv=8·2%). The corresponding figures for 30% moisture content are: EU: 77·2 ± 4·0 (5·2%); LU: 100·4 ± 7·4 (7·4%). The overall uniformity of either EU or LU model ages throughout the deposit, and the absence of an increase in age with stratigraphic depth indicates that the true age range within the deposit is small, and suggests that whatever the uptake history for the teeth may have been, it was uniform for all teeth throughout the deposit. One could alternatively suggest that there had been a gradual shift from early uptake to continuous uptake with increasing depth, which would also allow the calculated age of the teeth to increase slightly with depth. This concept is partially supported by the observation that the average U concentration in dentine increases with depth, as would be expected if the deeper teeth had been absorbing U for a greater fraction of the burial period than the teeth from higher stratigraphic levels. The difference in U content is small, however, and in the absence of confirmatory U-series data it is rather speculative to assume that the lower samples (95390–95392) are significantly older. Tests of the U uptake models for these teeth are in progress.

A further test of our age estimate can be made by comparison with the luminescence (TL, OSL) ages for the deposit obtained by Feathers & Bush (2000). They also find a strikingly small range of OSL ages, ranging between 65 and 75 ka (using a measured moisture content of 7%). These dates agree most closely with our EU ages for tooth enamel. In general, this agreement remains present if both the luminescence and ESR ages are calculated with increasing moisture content, with the former becoming somewhat older as values >20 M% are used. The departure occurs because the internal doses in the teeth make the M% dependence on age less significant than for the luminescence dates, which depend almost entirely on external doses. We also note that luminescence dates do not depend on a model of U uptake since the dose rate depends only on the U, Th and K content of the sediment itself which is assumed to have remained constant.

Conclusions

In conclusion, we find that nine of the ten enamel samples and subsamples give EU ages which agree closely for a given set of model conditions. For the most likely moisture content of 10%, the dates range between 64 and 75 ka, with an average of 70 ± 4 ka. If continuous (linear mode) uranium uptake had occurred, the ages would be about 28% older, averaging 88 ± 7 ka. Eventual U-series analyses of some of the teeth from this site should permit us to discriminate between these U uptake models.

An age of around 72 ka would place the deposition of the site within oxygen isotope stage 4 when sea level was significantly lower than at present, and would place the site at some considerable distance from the seashore. An age of 72 ka is consistent with the occurrence of a silcrete-dominated lithic assemblage—other silcrete-dominated assemblages are typically Howieson's Poort and this industry has been dated elsewhere to about this age at Klasies River Mouth (Thackeray, 1992). The linear uptake ages would place the industry at an age close to 90 ka, which is perhaps too old in comparison to other suggested ages for the industry, if we accept the widely held assumption that this distinctive industry was produced over a limited time range.

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