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Luminescence dating relevant to human origins

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SUMMARY

Luminescence dating provided the first direct and independent evidence that anatomically modern humans had a presence in western Asia earlier than is consistent with the 'regional continuity' model. The reliability of the result concerned, 92 (± 5) ka for burnt flints from Qafzeh Cave, is excellent and consistent with isochron analysis of the data. Flint dating has also confirmed palaeoenvironmental indications that the Mousterian industry in Europe was present somewhat earlier than the 100 ka limit previously accepted. Burnt quartz and unburnt sediment have also been important in Palaeolithic dating and the latter has a particularly high potential.

1. INTRODUCTION

(a) *Basis*

The two commonly used techniques of luminescence dating are thermoluminescence (TL) and optically stimulated luminescence (OSL), the latter being called optical dating by its originators (Huntley *et al.* 1985). For TL the dating signal is stimulated by heat whereas for OSL it is stimulated by light. With both, the signal is a measure of the population of electrons trapped at defects in the crystal lattice of the mineral being utilized (e.g. quartz, flint, feldspars); the build-up of this population is the result of continued exposure to the weak flux of nuclear radiation emitted by radioactive impurities in the sample and in the immediately surrounding sediment, together with a minor contribution from cosmic rays. The relevant radioactive impurities are ²³²Th, ²³⁵U and ²³⁸U together with their associated radioactive decay products, plus ⁴⁰K and ⁸⁷Rb, although the latter is of almost negligible importance. The relevant nuclear radiation consists effectively of α and β particles from within the sample, γ radiation from the burial surroundings (up to a distance of about 0.3 m) and cosmic ray mesons.

For the trapped electron population to be a useful measure of age it is essential that it was zero at some event in antiquity; it is that event which is dated. This 'zeroing' or 'resetting' can be by heating to upwards of around 400°C or by sufficient exposure to daylight. If pottery is under study then the event will have been its firing in the potter's kiln. In the case of burnt flint it will have been accidental falling into the fire or deliberate heat treatment. For aeolian deposits such as wind-blown sand and loess the resetting event will have been exposure to light during transportation or while lying exposed on the surface before being covered by further deposition. Waterborne deposits

will also have been reset, though not so effectively; the OSL technique is particularly advantageous for such sediment because with this technique the dating signal is stimulated only from highly light-sensitive traps whereas the TL signal comes from other traps too, some of which require hours of short wavelength 'bleaching' for their effective emptying; also there are some traps which are virtually immune to light exposure. This is in contrast to the 'brute force' zeroing that results from heating. Trapped electron populations of zero are also the case for newly-formed crystals e.g. stalagmitic calcite.

To translate the dating signal into calendar years two further quantities are required. First, for each sample studied it is necessary to measure the sensitivity, i.e. the signal resulting from a given dose of nuclear radiation; this is done by exposure to artificial radiation of known intensity. In this way the palaeodose, P , can be evaluated; this is the laboratory estimate of the dose that the sample must have received during antiquity for its dating signal to be equal to the observed value. Secondly, it is necessary to determine the dose-rate, D ; this is the dose per year that the sample has been receiving during its period of burial. One approach is to determine the concentrations of radioactive elements present in sample and soil by means of neutron activation analysis. Alternatively, direct measurement of the natural radioactivity is used, both within the laboratory and on-site. The cosmic ray contribution is obtained by calculation, having regard to thickness of overburden, altitude and latitude. In principle the age, A , is then obtained from the equation.

$$A = P/D. \quad (1)$$

The unit of dose is the gray (Gy). The age so obtained is directly in calendar years and it is independent of any other chronological technique.

In practice, equation (1) is deceptively simple and even with automation the derivation of a reliable result is complex and labour intensive; a dozen or so parameters need to be measured to make allowance for various subtle effects: accounts of these have been given elsewhere (Aitken 1985, 1990). It should be noted that in contrast to the radiocarbon and uranium-series techniques the radioisotopes involved have very long half-lives—in excess of 10^9 years; hence the dose-rate is basically constant (there may be small variations due to varying environmental conditions).

A measure of the trapped electron population can also be obtained by electron spin resonance (ESR) as discussed elsewhere in this volume. The three techniques are often collectively described as trapped electron dating. Recent reviews of luminescence techniques and applications have been given by Berger (1988), Wintle (1990), Zöller & Wagner (1990), Aitken (1989, 1992) and Valladas (1992); the solid-state mechanisms involved have been discussed, among others, by McKeever (1985).

2. RELEVANCE TO HUMAN ORIGINS

(a) *Burnt flint; burnt quartz*

Flint is a form of chalcedony, another being chert; in the context of dating the two are not usually distinguished, both being called flint. Its dating came into prominence with the publication by Valladas *et al.* (1987, 1988) of results for two caves in Israel: Kebara and Qafzeh. The result for the latter gave the first hard evidence that anatomically modern humans were present in that region some 90 ka ago, thus firmly rebutting any notion of evolution from Neanderthals; the cave at Kebara had contained a Neanderthal skeleton and the TL dates for the relevant levels indicated an age of close to 60 ka. These were subsequently supported by ESR. Recently another TL age indicating early arrival of modern humans in that region has been obtained at the cave of es-Skhul (Mercier *et al.* 1992). The reliability of the Qafzeh date is examined in § 3d.

Another contribution, recently reviewed by Valladas (1992), has been confirmation of palaeoenvironmental indications that the Mousterian industry in Europe was present somewhat earlier than the 100 ka limit generally accepted. Thus at Biache-Saint-Vaast in northern France, TL dating of six burnt flints gave an age of $175 (\pm 13)$ ka for the level in which two pre-Neanderthal skulls had been found; floral and faunal evidence indicated occupation during an interstadial of isotope Stage 6 (Huxtable & Aitken 1988a). Another site for which TL gave an earlier than expected age for a Mousterian (IV) level was at Abri Vaufrey in the Dordogne valley of France; four flints gave an average age of $120 (\pm 13)$ ka (Huxtable & Aitken 1988b); a similar age had previously been obtained for a stalactite fragment found in the same level (Aitken & Bussell 1982). The oldest site for which flint dates have so far been published is the Acheulian site of Maastricht-Belvédère in eastern Holland, the age obtained for the earliest occupation layer being $263 (\pm 22)$ ka (Huxtable & Aitken 1985).

Another silica mineral utilized is quartz and grains extracted from the clay of Palaeolithic fireplaces have been used for dating: ages of 31–36 ka have been obtained for the Lake Mungo fireplaces in Australia (Huxtable & Aitken 1977; Bell 1991). These are in satisfactory agreement with radiocarbon if allowance is made for that technique's probable underestimation by several thousand years (Mazaud *et al.* 1991).

(b) *Unburnt sediment*

Although burnt flint is an excellent material for TL dating, a severe problem on many sites is the scarcity of flints which are both well enough burnt and large enough for satisfactory processing. Hence the feasibility of dating the time of deposition of unburnt sediment vastly extends the scope of luminescence dating on Palaeolithic sites. An early study of sediments from Garrod's Tabun Cave was made by Bowman (1985). Although this was on polymineral samples there was indication that TL from quartz was dominating the signal, the problem of feldspar fading hence being alleviated. Bowman (1985) was reluctant to quote a date on account of dose-rate uncertainties associated with observed escape of radon from samples in the laboratory. Nevertheless it is interesting to note that using the data reported, the ages obtained (making approximation about radon loss; S. G. E. Bowman, personal communication) for two samples from Garrod's level D (the level below the female Neanderthal skeleton) were both about 160 ka, close to the linear uptake (LU) age of $166 (\pm 20)$ obtained for tooth enamel from that level by ESR (Grün *et al.* 1991). On the other hand TL dating (Mercier 1992) of 11 burnt flints from level D has given a preliminary average age of 250 ka. Of course in dating sediments in dark caves it is essential that the grains were carried in by wind (or water) rather than derived from the walls.

Another relevant study is that of Roberts *et al.* (1990), made as the first in a series concerning the time of initial human arrival on the Australian continent. Quartz grains extracted from sand on sites located on sand aprons at the foot of the Arnhem Land plateau were used and some dozen dates obtained at various depths in the profiles. There was satisfactory agreement with radiocarbon ages obtained for the upper layers of both sites, and at one of them three TL ages were associated with the lowest occupation level yielding stone artifacts; the authors suggest that human arrival in northern Australia occurred between 50 and 60 ka ago, somewhat earlier than previous indications obtained using the radiocarbon technique (which is of course at its limit around 40 ka).

In using TL for unburnt sediment the need to assess the effectiveness of resetting at deposition adds substantially to the work. The osL technique avoids this and highly encouraging results have been obtained in test programmes (e.g. Smith *et al.* 1990; Stokes 1992). An age of around 130 ka has been obtained (Stokes 1993) for quartz grains extracted from sediment of the lowest lake at Bir Tafawi (Egypt) in agreement with uranium-series and amino acid dating of ostrich eggshells.

(c) Stalagmitic calcite

Although TL can be used for dating this type of material in caves occupied by hominids back to 300 ka (e.g. at Caune de l'Arago, France, by Debenham & Aitken (1984)), in most contexts the heterogeneity of the calcite itself and of the surroundings means that reliable assessment of the external dose-rate is difficult. The uranium-series method does not have this drawback and hence is usually the preferred technique. The calcite date mentioned above for Abri Vaufrey was obtained for a small piece of stalactite which had fallen from the cave roof and become buried in sediment to a sufficient depth (0.3 m) for the external dose-rate to be reliably evaluated.

3. RELIABILITY**(a) General**

In addition to the concordances with other techniques noted above, accumulated evidence from a wide variety of samples and sites has established the general validity of the technique (see Ancient TL date lists, Bailiff, n.d.). Nevertheless, with a particular sample-type or a particular site there may be special sources of inaccuracy. Hence, however thorough the demonstration that a particular mineral, say quartz, gives accurate results on a 'good' site, it does not prove it will do so on a site where interfering factors, e.g. radioactive disequilibrium, are present; conversely failure in one particular site or with one particular mineral does not prove the whole technique to be invalid.

The dates mentioned in § 2 are mainly based on flint, which has the advantage of being a geochemically stable mineral. In addition to the results obtained by one of us (H.V.), there has been extensive flint dating by J. Huxtable at Oxford (see Ancient TL date lists); all these indicate burnt flint to be an excellent dating material. Additional comparisons with other techniques have been summarized by Valladas (1992). The oldest was for La Vigne Brun (France), yielding a flint date of 27 ka; this was in excellent agreement with the age obtained using burnt quartz and also with the average radiocarbon date for four charcoal fragments after making allowance for the probable 3 ka underestimation by the latter technique at the time period concerned (Bard *et al.* 1990).

Some additional general aspects are considered in the next two subsections and then there is specific discussion of the critical date for Qafzeh.

(b) The plateau test; residual signals

An advantage with the luminescence techniques is that the dating signal is more than a quantity, it has a characteristic shape too, and irregularities in this are indicative that all is not well. In the case of TL dating the signal is in the form of a glow-curve and comparison of the glow-curve shape observed for portions of an ancient sample 'as found' (the natural

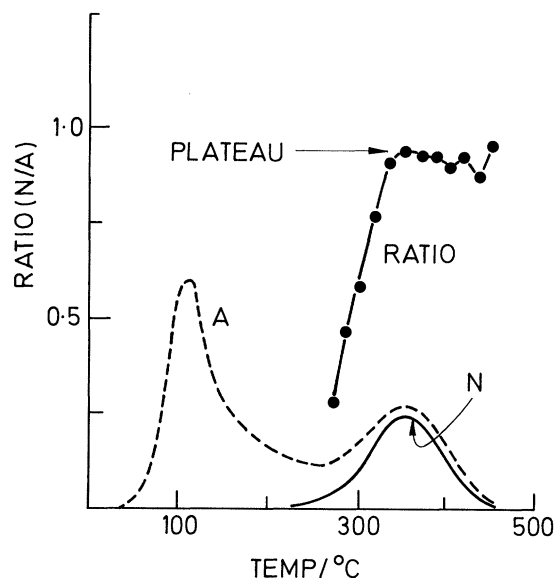


Figure 1. The plateau test. Curve N is the TL glow-curve for a flint that was well-burnt in antiquity and curve A is the additional TL observed from another portion to which a laboratory dose has been administered. The ratio (N/A) reaches a plateau in the glow-curve temperature region where the storage lifetime of the trapped electrons responsible for the TL is much greater than the age of the sample; at lower temperatures the TL is associated with electrons ejected from traps for which the storage lifetime is much shorter. It is the plateau value that is used for dating and in this example the ratio value (of 0.95) corresponds to the palaeodose being 0.95 times the laboratory dose administered. In practice a range of laboratory doses is used, with several portions for each dose point.

TL) with that for portions that have received laboratory irradiation allow the all-important plateau test to be made (see figure 1). Failure to pass this test can be for a variety of reasons: e.g. contamination, presence of 'spurious' TL (a parasitic signal indicative of poor measurement conditions), inadequate stability of the trapped electrons (i.e. their lifetime in the traps was not long enough to avoid leakage during the burial period). Failure can also be caused by insufficient heating at the resetting event in antiquity and given the difficulty mentioned earlier of finding enough well-burnt samples this is of particular relevance to flint dating (see figure 2). Because the plateau test is an intrinsic part of the measurement process there is no risk of dates being obtained from 'half-baked' flints. This eliminates the possibility of a flint date being erroneously too old on account of the dating signal containing a contribution of residual 'geological' TL.

However, the question of residual signal is of more serious concern in the TL dating of unburnt sediment; as mentioned earlier, in such application the residual signal may be appreciable and needs to be subtracted. Here again the plateau test is employed, although usually substantial complication is involved (see Mejdahl 1988). There is great region-to-region variability in this respect dependent on mode of deposition and intensity of daylight; at the Arnhem Land site in Australia mentioned in § 2b the straightforward

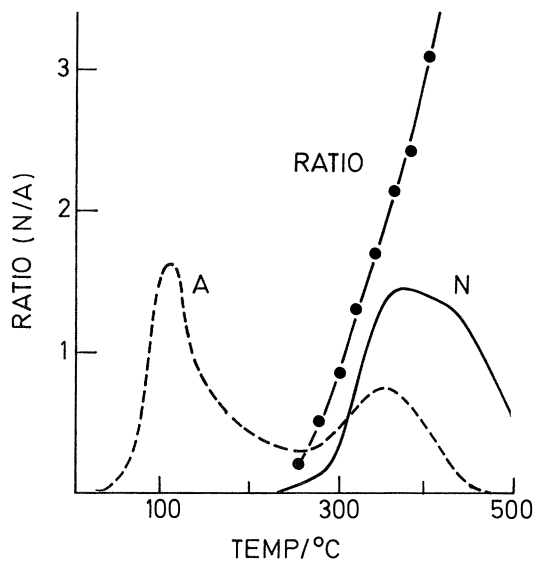


Figure 2. Failure to pass the plateau test. Curve N is the glow-curve from a flint that had not been burnt in antiquity; curve A represent the TL due to a laboratory dose administered after heating a sample of the flint to 500°C. Curve N has a different shape from that of figure 1 because in this unburnt case the trapped electrons have been accumulating during the very long time that has elapsed since the flint's formation. The data used are taken from Robins *et al* (1982).

For partially burnt flints the rise in the ratio is less steep but as is evident from the work of Melcher & Zimmerman (1977) there is no risk of even the semblance of a plateau with such flints, making calculation of a date impossible.

plateau was good, consistent with the observation that direct measurement of recently deposited sand at the site indicated a palaeodose of only one gray.

In the case of osL the corresponding necessary condition for reliability is a satisfactory 'shine-plateau': i.e. there should be no change in the paleodose calculated for successive intervals of exposure to the stimulating light.

(c) *Limitations on age range: lifetime; saturation; anomalous fading*

Stable retention of trapped electrons over the burial period is obviously a basic requirement in respect of the traps providing the dating signal, and as mentioned above a satisfactory plateau indicates that this condition has been met. However, it is useful to have some estimate of trapped electron lifetimes since this is a possible limitation in the age range of application. Laboratory 'kinetic' measurements indicate the following rough values (see Aitken 1985): for quartz and burnt flint, 10^8 years; for calcite, 10^6 years; for K-feldspar, 10^7 years. Thus we see that for the 100 ka time span, which is this volume's focus of interest, the lifetimes are adequate.

However, there is another limitation on the age range. This is that with continued exposure to radiation all available traps become filled. For quartz and flint the radiation dose at which this occurs is of the order of several hundred grays, and depending on the

level of radioactivity this corresponds to a limiting age of several hundred ka. Some types of these minerals saturate at lower doses and some at higher doses, so no strict limits can be laid down; it is a matter for investigation on each particular site. In general it seems that flint can reach further back than quartz.

For feldspars the saturation dose is much higher, a value of several thousand grays being applicable to K-feldspars for instance. However despite the adequate lifetime indicated by laboratory studies there is a tendency in some types for appreciable leakage of trapped electrons to occur. This is termed anomalous fading and whenever feldspars are used for dating special procedures and checks are necessary to exclude the possibility of interference by this malign phenomenon; this applies equally when a mixture of minerals is used: the luminescence from feldspars is usually dominant.

(d) *Qafzeh*

The date (Valladas *et al.* 1988) for this cave is based on 20 flints obtained from Mousterian layers within a 2.5 m section that had yielded anatomically modern humans ('Proto-Cro-Magnons'). According to the 'multiregional model' these humans evolved from Neanderthals and the date (92 ka) should have been more recent than the 60 ka date for Kebara. Hence the question at issue with the Qafzeh date is whether there was some factor that caused it to be erroneously

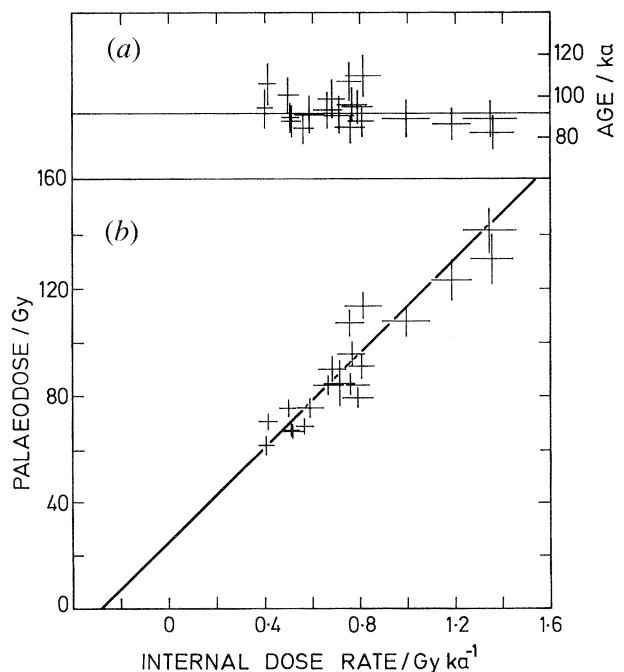


Figure 3. (a) (right-hand scale) TL ages for Qafzeh plotted against internal dose-rate values. The horizontal line represents the weighted average (as published by Valladas *et al.* (1988)). (b) (left-hand scale) Isochron plot of Qafzeh palaeodose values versus internal dose-rate values. The line is the least-squares fit, weighted for uncorrelated errors on both axes (York 1969); the slope indicates an age of $88 (\pm 9)$ ka and the intercept on the horizontal axis indicates an external dose-rate of $0.28 (\pm 0.09)$ Gy ka⁻¹.

too old. The most easily dismissed is the possibility that the flints were insufficiently heated at time zero; besides this being ruled out by the considerations of § 3*b*, it is also ruled out by the tight clustering of the individual dates (see figure 3*a*): the degree of insufficiency would vary from flint to flint.

Given that the paleodose evaluation followed well-established practice other possible causes of overestimation have to be sought: could the dose-rate have been higher during the millennia of burial than indicted by measurements made today? The impervious nature of flint makes it highly unlikely that there would have been any leaching away of internal radioisotopes (which contributed the dominant part of the total dose-rate) and even if it had occurred it would hardly have been the same from flint to flint. Direct evidence against leaching is that in the five flints for which fission-track mapping of uranium was carried out there was practically uniform distribution.

Turning to external dose-rate, albeit of lesser importance for this site according to the measurements reported, there are possibilities that cannot be dismissed *a priori*. One is that there might have been progressive leaching away of radioisotopes in the burial sediment. Another is that the moisture content of the sediment might have been lower during antiquity than at present: the presence of moisture causes attenuation of the flux of γ radiation reaching the sample. A third is that there could have been a change in the cosmic ray flux (which on this site is estimated to provide approximately half the external dose-rate) due to a change in the degree of shielding by rock.

If leaching had occurred a progressive increase in external dose-rate with depth would be expected. Reference to Table 1 of Valladas *et al.* (1988) clearly shows this not to be the case; in fact the extreme values of 0.22 and 0.25 Gy ka⁻¹ are easily contained within the quoted area limits (of ± 0.04 Gy ka⁻¹). Further, if leaching had occurred it would be expected that there would be disequilibrium in the uranium series; but measurements using α and γ spectrometry showed this not to be the case.

There is no direct evidence bearing on the other two possibilities but there is implicit indication in the reported data that any past variations in external dose-rate (and indeed in internal dose-rate too) have not been serious enough to distort the ages obtained. The internal dose-rates of the individual flints are widely spread: from 0.41 to 1.36 Gy ka⁻¹, unrelated to depth. Yet the ages obtained for flints of high internal dose-rate are not significantly different to those obtained for flints of low internal dose-rate (see figure 3*a*); if there was a significant systematic error in either dose-rate evaluation this would not be the case.

Similar evidence of reliability comes from isochron analysis, first used in TL dating by Mejdahl (1983) and demonstrated for ESR by Blackwell & Schwarcz (1992). This is possible because of the combined circumstances of a wide spread in the internal dose-rates, constancy of external dose-rate down the section, and the geological indications (see Valladas *et al.* 1988) that the sediment of the section accumulated

rapidly. Isochron analysis allows evaluation of average age without insertion of any value for the external dose-rate; it is only required that this latter is the same for all samples. Figure 3*b* shows its application: the age so evaluated is $88 (\pm 9)$ ka; the published age (92 ± 5) is consistent with this.

ESR dates for tooth enamel from the burial levels have also been obtained (Schwarcz *et al.* 1988) giving an early uptake (EU) age of $96 (\pm 13)$ ka and a linear uptake age (LU) of $115 (\pm 15)$ ka, the latter being favoured. These dates utilized the external dose-rate that was evaluated for the TL but in the case of ESR this is the dominant contribution to the overall dose-rate. Hence the two techniques are not strongly interdependent as far as dose-rate is concerned, and given the different nature of the crystal structures in which the trapped electrons were accumulated the ESR result can be considered as independent confirmation of the site's great age. The fact that the favoured ESR age of $115 (\pm 15)$ ka is somewhat greater than the TL age (92 ± 5 ka) may be due to underestimation of the external dose-rate. The isochron analysis of the TL dates (figure 3*b*) indicates an external dose-rate of $0.28 (\pm 0.09)$ Gy ka⁻¹ whereas the values used (for both TL and ESR) were in the range 0.22–0.25 Gy ka⁻¹. Because of the dominance the external dose-rate in the case of ESR, use of the isochron value would tend to move the ESR result into agreement with the TL.

Finally, it is to be noted that the scatter in the twenty individual TL ages corresponds to a standard deviation of only 8%; this is very close to the predicted random ('statistical') error limits quoted for the individual dates. Hence these twenty ages form a remarkably coherent group, thus giving further strong support to the validity of the result. Such coherence is not always the case: for instance the six individual TL ages on which the average of 119 ka for es-Skhul (see § 2*a*) are based show a much wider scatter, the standard deviation being around 18%. This is indicative of less favourable circumstances, as was recognized by Mercier *et al.* (1992) who quoted predicted error limits of ± 18 ka. Thus in making archaeological interpretation of these results for the presence in the region of anatomically modern humans, it is appropriate to put emphasis on the TL result for Qafzeh.

5. SUMMING UP

Luminescence techniques allow the independent dating, directly in calendar years, of burnt flint, burnt quartz and calcite, as well as unburnt wind- and water-borne sediment; for the latter it is advantageous to use the OSL technique. Consideration of the burnt flint TL data for Qafzeh underlines the reliability of that result, one of several which are critical for our understanding of the origin of modern humans.

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