The Rb contents of the K-feldspar grains being measured in optical dating

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(Received 9 January 2001 ; in final form 22 October 2001)

Abstract: *A value of 400 ± 100 $\mu g \cdot g^{-1}$ for the rubidium content of K-feldspar grains is recommended for use in the dose-rate calculation in the absence of more detailed information. The corresponding dose rate is 0.15 ± 0.04 $Gy \cdot ka^{-1}$, multiplied by the absorbed energy fraction. This fraction is < 1, even for sand-sized grains, thus calculations of this fraction are needed.*

Introduction

Following suggestions by Mejdahl (1983, 1985), sand-sized K-feldspar grains are often separated and used for dating. A large fraction of the dose rate in such grains arises from the β particles emitted by the potassium within the grains, and Huntley and Baril (1997) earlier recommended the use of a value of 12.5 ± 0.5 % K for calculating the dose rate from this potassium. This recommendation was based on the measurements of the K contents of separated grains, scanning-electron-microscopy element maps to determine the fraction of the grains that were actually K-feldspar, and the observation that grains with the highest K contents gave the most luminescence (Prescott and Fox, 1993; Spooner, 1992).

Mejdahl (1987) pointed out that the β particles emitted by the Rb within the K-feldspar grains also make a significant contribution to the dose rate, and this must also be evaluated. Here we address the question of what Rb content should be assumed in the absence of detailed information.

Rb and K have the same outer electronic structure and very similar ionic radii, and consequently accompany each other during geochemical processes, just as different isotopes of the same element do. Their ionic radii are relatively large and this restricts the lattice sites which they can occupy. The result is that the Rb/K ratios are about the same for different minerals formed from a given magma. With this in mind one could imagine that there would be a universal Rb/K ratio for all rocks and sediments. It is not so.

Smith and Brown (1988, pp.348-353) summarized a large number of Rb analyses of a wide range of feldspars. They observed that K/Rb ratios vary from 4 to 17,000. The reader is referred to this book for detailed summaries for feldspars from various rock types. Our particular interest is restricted to feldspars with high K contents, and for these most Rb contents lie in the range 100-3000 $\mu g \cdot g^{-1}$, although values outside this range occur. Mejdahl (1987) reported K and Rb contents for 27 samples of feldspars extracted from pottery, burnt stone and sediments; he found a good linear correlation and suggested using this for estimating Rb contents. For those samples for which the K content was above 10%, the Rb contents were in the range 300-600 $\mu g \cdot g^{-1}$, the average being about 400 $\mu g \cdot g^{-1}$.

Here we use a different approach to the problem. The method used is similar to that used by Huntley and Baril (1997) to obtain the K-content recommendation, and the samples used are the same ones. In Table I the first four columns list the sample names, K contents of the separated grains, the fraction of the grains that were found to be K-feldspars using scanning-electron-microscopy element maps, and the deduced K contents of the actual K-feldspar grains. These are the same data that appeared in Huntley and Baril (1997).

The next column lists the Rb contents of the separated grains determined by neutron activation analysis. Next we assume that all the Rb is actually in the K-feldspar grains, i.e. that it is not in the quartz or plagioclase grains that are also present in the separates, and use the fractions of column 3 to calculate the Rb contents of the K-feldspar grains. The results are in the column labelled 'Rb in K-feldspar grains'. Some idea of the reproducibility of the technique can be obtained by comparing results from samples which are expected to

sample	K in separated grains wt.% (± 5%)	K-feldspar fraction	K in K-feldspar grains wt.%	Rb in separated grains $\mu g \cdot g^{-1}$	Rb in K-feldspar grains $\mu g \cdot g^{-1}$	K in whole sample % (± 5%)	Rb in whole sample $\mu g \cdot g^{-1}$	11 % x sample Rb + sample K $\mu g \cdot g^{-1}$
TTS	4.8	0.44 ± 0.05	10.9 ± 1.2	116 ± 2	264 ± 30	1.08	25 ± 2	255
TTS3	9.05	0.68 ± 0.05	13.3 ± 1.0	158 ± 4	232 ± 18	1.02	28 ± 2	302
CBTS2	9.6	0.68 ± 0.05	14.1 ± 1.0	177 ± 3	260 ± 20	1.01	28 ± 2	305
FHTS-3	4.7	0.35 ± 0.04	13.4 ± 1.7	93 ± 8	266 ± 38	1.12	25 ± 3	246
KHTS-1	5.7	0.46 ± 0.04	12.5 ± 1.2	93 ± 6	202 ± 22	1.11	20 ± 1	198
KHTS-2	3.9	0.28 ± 0.04	13.4 ± 1.7	71 ± 3	254 ± 38	1.24	22 ± 3	195
SW6-01	7.6	0.55 ± 0.05	13.8 ± 1.2	225 ± 4	409 ± 38	1.36	44 ± 2	356
SAW94-32	6.2	0.47 ± 0.05	13.2 ± 1.4	165 ± 4	351 ± 38	1.50	47 ± 2	345
SAW94-37	8.3	0.64 ± 0.04	13.0 ± 0.8	236 ± 4	369 ± 24	1.48	42 ± 2	312
SAW94-62	2.4	0.37 ± 0.05	6.5 ± 0.9	54 ± 3	146 ± 21	1.70	72 ± 7	466
MELVL93-5	10.7	0.74 ± 0.04	14.5 ± 0.8	348 ± 5	470 ± 26	2.19	136 ± 4	683
CPIW	6.0	0.44 ± 0.05	13.6 ± 1.5	233 ± 4	530 ± 61	1.03	39 ± 2	417
TAG2	7.9	0.60 ± 0.06	13.2 ± 1.3	176 ± 4	293 ± 30	1.25	44 ± 2	387
CTL2	2.3	0.15 ± 0.04	15.3 ± 4.0	46 ± 3	307 ± 84	1.55	39 ± 2	277
DY24	11.5	0.93 ± 0.02	12.4 ± 0.3	374 ± 7	402 ± 12	2.28	80 ± 2	386
SN30	11.9	0.97 ± 0.02	12.3 ± 0.3	380 ± 20	392 ± 22	0.57	23 ± 1	444
SN55	11.4	0.96 ± 0.02	11.9 ± 0.3	370 ± 20	385 ± 22	0.59	23 ± 2	429
SN4d	3.4	0.28 ± 0.05	12.1 ± 2.2	109 ± 3	389 ± 70	0.52	19 ± 1	402

Table I: *Column 6 lists Rb contents of the sand-sized K-feldspar grains in grain separates used for optical dating, calculated from measured Rb contents of the separates and the fraction of those grains that are K-feldspars. The last column lists alternative estimates based on the Rb contents of the bulk samples, made as described in the text. The first 6 samples are from tsunami-laid sands from British Columbia and Washington State (Huntley and Clague, 1996; Baril, 1997). Samples SW6-01, SAW94-32 and SAW94-37 are from interdune and dune sands from the Great Sand Hills region of Saskatchewan (Wolfe et al., 2001). Sample SAW94-62 is loess from the base of the Cypress Hills nearby. Sample MELVL93-5 is from the Melville Gap site, California, U.S.A. (Rockwell et al., 2000). Samples CPIW and TAG2 are from a sand wedge and a fluvial deposit respectively from the Mackenzie River delta. Sample CTL2 is from the Coutlee section near Merritt, British Columbia. Sample DY24 is from an aeolian deposit on a terrace by the Lena River near Yakutsk, Siberia. The SN samples are from Sandy Neck, Cape Cod, Massachusetts, U.S.A. (van Heteren et al., 2000)*

be similar; these are: samples TTS and TTS3, KHTS-1 and KHTS-2, SAW94-32 and SAW94-37, and the three SN samples.

Most of these Rb estimates lie in the range 200-400 $\mu g \cdot g^{-1}$. Our average value is lower than that found by Mejdahl (1987), presumably reflecting differences in our samples. Combining our two data sets, the overall picture that emerges is that a value of 400 ± 100 $\mu g \cdot g^{-1}$ Rb covers most of the range observed at ± 2σ, and this is the value recommended for the dose-rate calculation if the actual Rb content is not available. With the assumption that all the β energy is deposited within the grains, the dose rate for this concentration of Rb is 0.15 ± 0.04 $Gy \cdot ka^{-1}$, calculated using the conversion factor of Adamiec and Aitken (1998).

Mejdahl (1987) states that the assumption that all the β energy is absorbed within the grains is valid for grains of 100 μm diameter and larger. This is in contradiction to the equation of Bell (1979, equation 8) from which one deduces that for a 100 μm diameter grain only 57% of the β energy is absorbed by the grain. The average β energy is 0.082 MeV, and the corresponding range is 0.013 $g \cdot cm^{-2}$, or about 50 μm in feldspar. It is clear that a significant fraction of the energy from β particles originating in a 100 μm diameter grain will be deposited outside that grain, and Mejdahl's statement is untenable. There is a clear need for a proper calculation of the absorbed energy fractions as was done by Mejdahl (1979) for the β particles from K, U and Th.

The last three columns of Table I show the results of an attempt to find out whether or not an estimate of the Rb content can be made using a much simpler method. We return to the idea of a fixed K/Rb ratio for all the minerals from a particular igneous source. The K/Rb ratio of the feldspar grains should then be the same as the K/Rb ratio of the bulk sample. Fig.1 shows a comparison of these two ratios for the present samples. While the expected correlation is found, there is a clear tendency for the K/Rb ratios of the separated grains to be higher than in the bulk sample. The Rb contents of the grains are thus a little lower than would be obtained using the assumption that the ratios were the same. We deduce that the best estimate of the Rb within the K-feldspar grains is calculated as (11 %)(Rb content of the sample ÷ K content of the sample), and these figures are shown in the last column of Table I. A comparison of these Rb content estimates with the determined Rb contents is shown in Fig.2. These estimates seem surprisingly good in view of the assumptions made, and are better than the fixed value for the Rb content suggested above. The figure of 11 % was simply chosen to provide the best fit to the data.

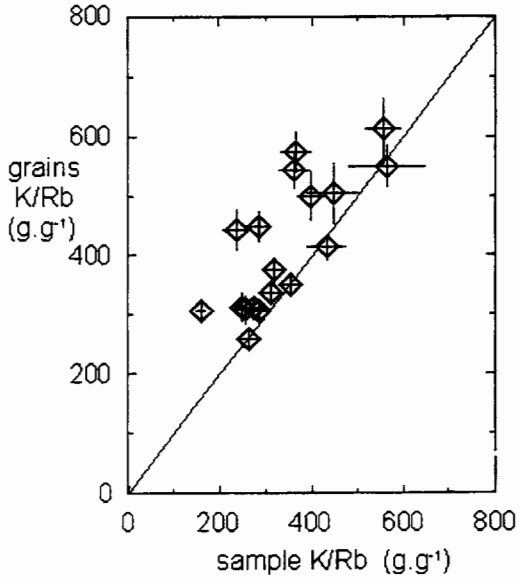

Figure 1.
Comparison of the K/Rb ratio of the separated grains with the K/Rb ratio of the bulk sample. There is a clear tendency for the former to be higher than the latter. The solid line represents perfect agreement.

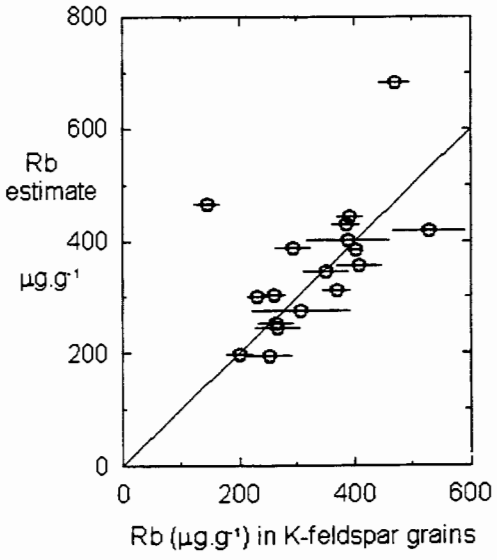

Figure 2.
Comparison of Rb contents of K-feldspar grains as determined by:
abscissa: measurements of Rb contents of the grains themselves
ordinate: an estimate calculated as Rb content of the bulk sediment x(11% ÷ K content of the bulk sediment).
The solid line represents perfect agreement.

If the sediment of interest is derived from a single igneous source, this procedure should yield the Rb contents of all the grains. If, on the other hand, the sediment is derived from two or more sources this procedure would yield an average Rb content, but not necessarily the Rb content of any particular grains, and in particular not necessarily the Rb content of the particular size of K-feldspar grains selected for dating. We note finally that the luminescence being measured is dominated by a small number of the thousand or more grains of an aliquot, and it is possible that these grains have Rb contents that are either higher or lower than typical Rb ones. This is a problem that needs to be pursued.

Grains were separated and prepared for analyses by M.R.Baril, G.O.Morariu and B.W.-L.Ong. Neutron activation analyses were performed at the Slowpoke-2 Facility, Royal Military College of Canada, Kingston, Ontario. H.P Schwarcz is thanked for his thoughtful comments. The Natural Sciences and Engineering Research Council provided financial support.

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Rewiever

H.P. Schwarcz

Alpha sensitivity determination in quartzite using an OSL single aliquot procedure

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(Received 19 October 2001 ; in final form 10 December 2001)

Introduction

In the process of dating burnt stone artefacts, the determination of the alpha sensitivity is of paramount importance especially when the radioisotopic contents of the pieces under study are high (typically > 1 ppm), since in such cases the contribution of the alpha dose rate to the total dose rate is not negligible. This parameter is also known to vary from sample to sample, as in the case of burnt flints dated by TL (see for instance Mercier et al., 1995), and its value is needed for each sample.

Up to now, the OSL technique has been rarely used for measuring this parameter and in cases where it has been done, it concerned mainly quartz grains extracted from sediments (Rees-Jones, 1995). Here we report alpha sensitivity measurements, using TL and OSL, made on burnt quartzite pebbles, which we wish to date by OSL. It will also be shown that an OSL single aliquot method can be used in order to determine this parameter for each disc, after its equivalent dose (ED) has been measured.

Alpha sensitivity : S-α

The alpha sensitivity under consideration (S-α) (Valladas and Valladas, 1982) is defined as the ratio of the relative alpha and beta sensitivities :

$$S\text{-}\alpha = \frac{L\alpha \,.\, D\beta}{\phi\alpha \,.\, L\beta}$$

where Lα and Lβ are the luminescence intensities induced by the artificial alpha integrated flux φα (number of α/cm^2) and the artificial beta dose Dβ (Gy), respectively. The equivalent beta dose rate $D\alpha_{equ.\beta}$ to be considered in the age equation is the product of the S-α value and the alpha flux rate, which depends on the specific U and Th alpha fluxes (19400 and 5377 respectively; Valladas, 1988) and on the radioisotopic contents of the sample[1].

[1] Thus, S-α can also be expressed to a first approximation as:

$$S\text{-}\alpha = k \,.\, \frac{[U].\, d_{sU} + [Th] \,.\, d_{sTh}}{[U] \,.\, \phi_{sU} + [Th] \,.\, \phi_{sTh}}$$

where k is the usual k-value (Zimmerman, 1971) and d_{sU} and d_{sTh} are the specific alpha doses for U and Th, respectively.

$$D\alpha_{equ.\beta} = S\text{-}\alpha \,.\, \{ \eta_U \,.\, [U] \,.\, \phi_{sU} + \eta_{Th} \,.\, [Th] \,.\, \phi_{sTh} \}$$

where units are :

S-α : Gy / α/cm^2

ϕ_{sU}, ϕ_{sTh} : number of α/cm^2 / p.p.m. / y

[U], [Th] : p.p.m.

and where η_U and η_{Th} are two correcting factors that are close to unity, the exact values depending on the alpha particle energies inside the sample under irradiation.

This definition assumes that the luminescence signals vary linearly with the beta dose and the alpha integrated flux. For all the samples we studied, this last assumption was true for OSL signals induced by alpha integrated fluxes up to 40.10^8 α/cm^2 (see sample CAR 7 in Fig.1a as an example), a value which is generally higher than the natural alpha integrated flux received by the sample since it was last heated. However, the OSL beta growth curve can exhibit a strong saturation behaviour as in the case of sample CAR 7, which contrasts with the beta induced TL signals which increase linearly up to 250 Gy (Fig. 1 b). Consequently, a correction of the OSL S-α value is necessary for samples exhibiting this behaviour in order to evaluate the mean S-α value they experienced during the burial time.

Samples and equipment

The samples used were archaeological burnt lithics (quartzite pebbles) from 3 Upper Palaeolithic sites in the Côa valley (Portugal): 4 from Cardina (CAR4, CAR5, CAR7, CAR8), 3 from Olga Grande Sul (OGS1, OGS2, OGS5) and 1 from Quinta da Barqua (QBS2). Their TL S-α values had been measured during a prior dating study (Valladas et al., 2001; we note here that the units for S-α in Table 1 of that paper should have been shown as μGy / 10,000 α / cm^2), in which fine grains previously heated at 350°C for 90 minutes were deposited on stainless steel discs, then irradiated either with a $^{90}Sr/^{90}Y$ beta source delivering 0.134 Gy/sec or with a ^{238}Pu source having a flux of 2.279 x10^6 α/cm^2/sec The TL signals were measured with a home-made automatic reader (Valladas et al., 1994) equipped with a blue-UV filter

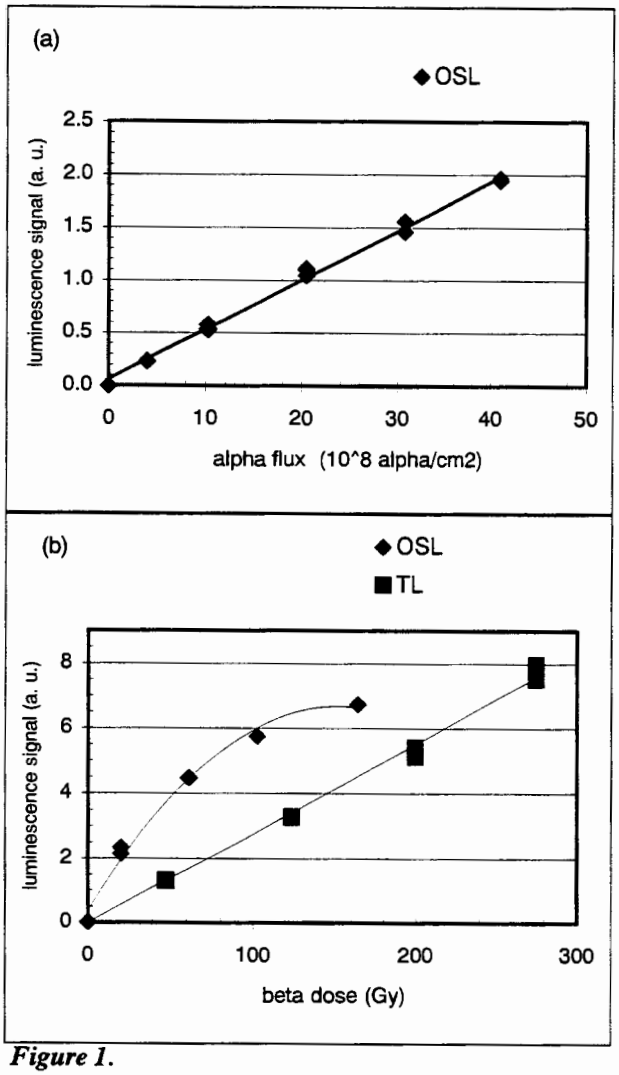

Figure 1.
(a) Sample CAR7: the linearity of the growth curve as a function of the integrated alpha flux is typical of what is observed when the luminescence signal is measured using the OSL and TL techniques.
(b) TL and OSL beta regenerated growth curves for the same sample. The TL and OSL growth curves behave differently, the TL one showing greater linearity.

(MTO 380 nm) and normalised by irradiating all discs with a fixed beta dose.

The OSL measurements reported here were performed on a Risø TL/OSL-DA-15 apparatus. The stimulation was done with a broad-band blue and green light (420-550 nm) filtered from a halogen lamp (Bøtter-Jensen and Duller, 1992). Luminescence was measured by a Thorn-EMI 9235QA photomultiplier tube using 3 Hoya U-340 filters. Beta irradiation came from a $^{90}Sr/^{90}Y$ source delivering 0.165 Gy/sec (attached to the Risø reader) and alpha irradiation from the ^{238}Pu source mentioned above.

Sample preparation and measurement procedure

Two sets of fine grains were prepared to evaluate the influence of pre-treatment on the OSL S-α values: one containing recently heated grains (set H) and another bleached grains (set B).

The preparation of set H was very similar to the one used in TL: the pebbles were crushed and sieved in order to obtain a < 100 µm granulometric powder which was heated at 350°C for 90 minutes in a furnace, then washed with hydrochloric acid, pure water, ethanol and acetone. Fine grains were deposited in acetone on stainless steel discs according to Zimmerman's protocol (1971). Five discs were prepared for each sample.

The same procedure was applied to set B, except that unheated fine grains were bleached for 100 sec at 125°C in the Risø reader.

Both sets were exposed to the alpha source at the integrated fluxes reported in column 1 of table 1. For comparison, integrated fluxes experienced by the samples during their period of burial (estimated from the TL ages and radioisotopic contents) are given in column 2.

sample	(1)	(2)
CAR4	41.02	26.20
CAR5	41.02	5.11
CAR7	41.02	28.21
OGS1	41.02	4.48
OGS2	41.02	3.98
OGS5	54.70	15.84
QBS2	30.08	5.99

Table 1.
(1) : integrated alpha flux (10^8 alpha/cm2) used for S-alpha determination
(2) : integrated alpha flux (10^8 alpha/cm2) experienced by the sample during burial calculated from age estimation and U, Th contents

We used a standard SAR protocol (Murray and Wintle, 2000) to determine the S-α value. For each aliquot, 7 cycles were measured, each one consisting of : β dose (= 0 for cycles n°1 and n°2), preheat at 220°C for 10 sec, OSL measurement for 100 sec at 125°C, test dose, cut-heat at 160°C and OSL for 100 sec at 125°C. The percentage of thermal transfer was estimated during cycle n°2. Increasing regenerative doses allowed us to build the beta growth curve; the dose of cycle n°7 was identical to the one of cycle n°3 to check the efficiency of the sensitivity correction. Using set B of sample CAR7 as an

example (Fig. 2), the beta doses for the successive cycles were 0, 0, 22, 65, 108, 173 and 22 Gy and the test dose was 7 Gy.

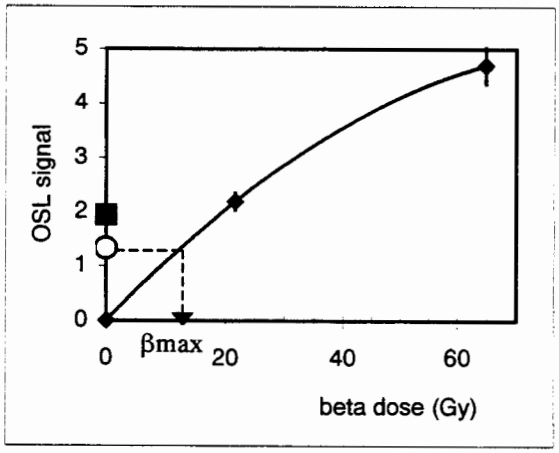

Figure 2.
SAR protocol applied to sample CAR7 (set B). The plot shows the region up to 65 Gy. The square represents the alpha induced OSL signal and the diamonds the beta regenerated signals.
The open circle represents the signal that would have been observed if the integrated alpha flux had been equal to the estimated archaeological alpha flux ($\phi_{arch.}$). As defined, β_{max} is the equivalent beta dose corresponding to this flux, giving a maximum value of S-alpha :
$S\text{-}\alpha_{max.} = \beta_{max} / \phi_{arch.}$. The mean value $S\text{-}\alpha_{corr.}$ corrected for non-linearity is then obtained by integrating or, simply, by averaging :

$$S-\alpha_{corr.} = (1/100)\sum_{n=1}^{n=100}\{\beta_n /(n.\Phi_{arch.}/100\}$$

where β_n is the dose corresponding to an integrated flux of : $n . \phi_{arch.}/100$

To correct for the non linearity of the regenerative curve, we first calculated by proportion the OSL signal which we would have observed if the integrated alpha flux had been equal to the estimated archaeological alpha flux ($\phi_{arch.}$) and deduced the corresponding beta dose (β_{max}) by using the regenerative beta growth curve. In the last step of the calculation, the S-α value was computed by averaging the values calculated between 0 and β max. The obtained S-α value is then slighlty dependent on the estimated age and an iterative procedure should sometimes be employed.

For the present sample under consideration (set B of CAR7, Fig. 2), $\phi_{arch.} = 28.2 \times 10^8$ α/cm^2 and β_{max} = 12.6 Gy. The corrected value $S\text{-}\alpha_{corr.}$ is then = 4.47μGy/ 10^3α/cm^2.

Results of comparisons

The mean OSL S-α values obtained after bleaching (set B) or after heating (set H), and the TL S-α values, are plotted in figure 3. For all the samples under study, in view of the associated errors, the two OSL S-α values are indistinguishable. However, the TL values tend to be higher than the OSL ones (20% higher for CAR4, CAR7, CAR8 and OGS2, 40% higher for OGS5 and 190% higher for QBS2). The reason for this difference is not clear. It may be due to the fact that traps and luminescent centres involved during the two kinds of measurements are not strictly identical.

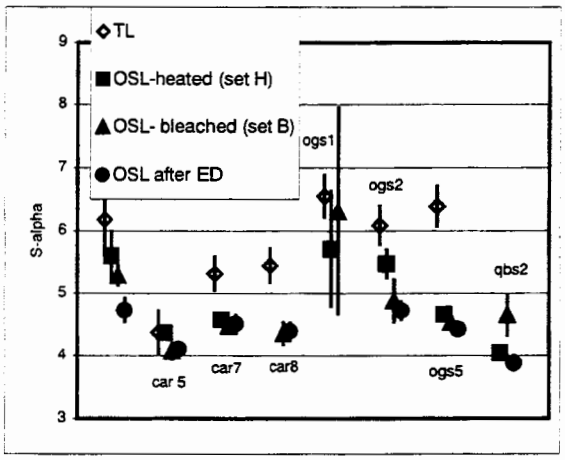

Figure 3.
TL or OSL S-α values (in μGy/ 1000alpha/cm^2) obtained after different sample pre-treatments. In view of the uncertainities, the OSL values measured for each sample are all very similar. On the other hand, the TL values tend to be higher : they are almost 20% higher for CAR4, CAR7, CAR8 and OGS2, 40% higher for OGS5 and 190% higher for QBS2. The TL S-α value for QBS2 (12.1 ± 1.2 μGy/ 1000 alpha/cm^2) is not plotted for clarity.

We have already seen that the S-α measurements make possible the determination of the alpha sensitivity from a single aliquot, which is an appreciable advantage over protocols using k or a-values requiring several aliquots. Moreover, the good agreement between the two sets of OSL S-α values indicates that the alpha sensitivity is independent of the past history of the sample. Consequently, it should be possible to use the same disc to determine first the ED and then the S-α value.

To test this hypothesis, another set of 5 fine grain discs were prepared for all the archaeological samples and their EDs were measured using the SAR protocol. Subsequently, each disc was alpha irradiated and its OSL S-α measured with the same SAR protocol. For each sample, the mean OSL S-α

value is reported in fig. 3. As expected, it does not appear to be significantly different from the values of sets H and B: all measurements can thus be performed on a single aliquot.

If we consider sample CAR7 once again, the OSL S-α values measured for 5 discs after ED determination were 4.68, 4.53, 4.55, 4.42 and 4.39, giving a mean of 4.51 μGy/ $10^3\alpha/cm^2$. Considering the U and Th concentrations of this sample (1.475 p.p.m. and 12.086 p.p.m., respectively) and the η_U and η_{Th} coefficients specific to the alpha source used, the beta-equivalent alpha dose rate ($D\alpha_{equ.\beta}$ is then :

$4.51 \times 10^{-3} \times \{ 0.87 \times 1.475 \times 19400 + 0.91 \times 12.086 \times 5377 \} = 379$ μGy /y .

Conclusion

For the quartzite pebbles under study, the OSL S-α sensitivity seems to be independent of the pre-treatment of the sample. To our knowledge, this is the first time that an experimental procedure allows for the ED and alpha sensitivity determinations to be made on a single aliquot. This might be particularly interesting for the dating of both sediments and small samples like pottery sherds.

Acknowledgements

We would like to thank Richard 'Bert' Roberts for his detailed review of the article and his fruitful comments.

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Reviewer

Richard Bert Roberts

Confirmation of backscattered beta dose enhancement rates based on single aliquot regeneration (SAR) analysis of quartz sand and silt

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(Received 1st August 2001 ; in final form 6 December 2001)

Introduction

In luminescence dating applications numerous factors contribute to systematic uncertainties in age estimation. Many of these parameters are associated with artificial irradiation within the laboratory, necessary for establishing the luminescence sensitivity of the given dosimeter. In dating applications this is now routinely achieved using calibrated 5-100 mCi $^{90}Sr/^{90}Y$ beta radiation sources, typically attached to automated TL/OSL reader devices (Bøtter-Jensen et al., 2000). While accurate cross-calibration of sources, either to known gamma doses or to other beta sources, is readily achievable, such exercises have previously been hampered in some instances by a lack of precision. This lack of precision is derived from a number of factors including (from Murray (1981) with additions):

- Contrasts in attenuating and backscattering of phosphor materials and substrates used.
- Difficulties imposed in off-plate calibrations (including disc orientation and possible source inhomogeneity).
- The size and shape of the phosphor grains.
- Thermal lag effects in thermoluminescence (TL) measurements.
- Inter-aliquot normalization where multiple aliquot approaches are employed.
- For some phosphors (e.g., quartz) problems associated with thermoluminescence analysis include thermal quenching, low temperature TL peak instability, and dose-dependent and other forms of sensitivity change.

By far the majority of the correction factors relating to dose rate contrasts between varying grain sizes and/or backscatter dose enhancements were derived empirically during early phases in the development of luminescence methods, principally employing CaF_2 as a high sensitivity TL phosphor (e.g., Wintle and Aitken, 1977; Murray, 1981; Aitken, 1985). The combination of optical dating and single aliquot regeneration (SAR) approaches provide radically improved means by which high precision estimates can be obtained for many of these important conversion factors. Somewhat surprisingly, these factors have not yet been systematically re-measured using such approaches.

It is the purpose of this note to describe two simple experiments which sought to re-evaluate the substrate dependent backscatter enhancement of beta dose from a $^{90}Sr/^{90}Y$ source. We tested both sand-sized (125-180 μm) and fine silt (4-11 μm) grain sizes; the sand comprising a gamma irradiated, annealed beach sand, and the fine silt consisting of a well mixed natural fine grained quartz sample (Ingram, 2001). Our results confirm the existing Al/stainless steel ratio (0.86) which was initially estimated as part of a larger experiment using 90-125 μm grains of CaF_2, and four contrasting backscattering materials (perspex, Al, stainless steel Pb) of infinite thickness with respect to the incident beta particles (Murray, 1981).

Samples Used and Sample Preparation

Ideally, either a geologically-stable signal from a naturally occurring phosphor, preferably the sample dosimeter used, or an artificially, uniformly irradiated phosphor should be employed to test for contrasts in dose backscattering from differing sample substrates.

In our study we used both the Equivalent Dose (D_e) from fine-grained (4-11 μm) quartz extracted from a deep sea core collected from the Indian Ocean (Ingram, 2001) and an annealed beach sand (125-180

μm) sample which had been artificially gamma irradiated to test for backscatter dose attenuation. The beach sand was collected from the contemporary upper (high tide) shore face of Eastbourne beach, UK and was annealed at 650°C for 50 minutes. The fine-grained aeolian quartz in the deep sea sediment samples is primarily derived, via long-distance aeolian transportation, from the Arabian Peninsula, and is well mixed at deposition by bioturbation processes, occurring in the upper few cm of the sediment column.

The beach sample was collected and underwent standard separation procedures for the refinement of fine sand (90-150 μm) sized quartz (Stokes, 1994). The fine silt sample was collected from a core repository in film canisters in reduced light conditions. The edge of the sample exposed to light during collection was removed in the laboratory. The gamma irradiation took place at the National Physical Laboratory (Teddington, UK) where the prepared quartz fraction was subjected to a total dose of 7.46 Gy using the Hotspot 800 ^{60}Co gamma source. The sample was then deposited as a monolayer on 4 aluminium and 4 stainless steel discs of a standard size (dia. 9.9 mm, thickness 1 mm).

The laboratory procedures to isolate fine-grained quartz from the bulk samples for OSL dating were adapted from standard practice as outlined by Aitken (1998). The bulk samples were sequentially treated with dilute hydrochloric acid to remove carbonates, hydrogen peroxide to remove organic matter and a dilute sodium oxalate solution to deflocculate the grains. To remove unwanted feldspars the samples were treated with fluorosilicic acid (Rees-Jones, 1995). The 4 –11 μm grain size fraction was isolated from the quartz fraction by settling in acetone according to Stokes' Law of settling velocities. The fine-silt suspension was then deposited, in acetone, onto aluminium and stainless steel discs of a standard size.

The purity of both samples was tested by infra-red stimulation – no significant IRSL above background was observed.

Single Aliquot Regeneration (SAR) Procedure

The Equivalent Dose (D_e) of the refined quartz was analysed using the single aliquot regeneration (SAR) protocol (Murray and Wintle, 2000). Measurements were made using a RISO TL/OSL DA-15 reader fitted with a blue (λ= 470 nm) diode array, and a calibrated ^{90}Sr/^{90}Y beta radioactive source and Electron Tubes type 9235QA photomultiplier. The natural signal was measured as initial (0-1s) luminescence intensity from which background levels (measured after 11-15 seconds exposure) was subtracted. The same aliquot was irradiated with successively increasing laboratory radiation doses and the regenerated OSL signals measured as for the natural to generate a 'growth curve'. A pre-heat of 260C for 10 seconds was employed prior to all measurements. Sensitivity changes induced by multiple irradiations were monitored and corrected via the measurement of OSL resulting from a test dose (10 Gy) and pre-heat (quick heat to 200C, no hold) in the SAR analysis. The D_e required to produce the natural OSL signal was interpolated from the growth curve. The robustness of the SAR analysis was tested in all cases via the re-dosing and measurement of the initial regeneration dose at the end of the experiment, producing a so-called 'recycling ratio'(Murray and Wintle, 2000).

Results

Our analysis has confirmed the efficiency and precision of the SAR approach (Tables 1 & 2, Figure 1). In all cases the recycling rations fell within 10% of 1.0. Our study confirms the great potential of the SAR procedure for reducing uncertainties associated with some conversion factors and ratios crucial to accurate luminescence ages. Grouping D_e estimates into averages for both aluminium and stainless steel, and sand-sized (Table 1) and fine silt-sized (Table 2) dosimeter fractions and calculating standard errors for those mean values results in substrate backscattering ratios which are known to better than 3%.

The resulting stainless steel to aluminium D_e ratios are 0.82±0.01 and 0.84±0.03 for sand and fine silt size fractions. These respectively correspond to backscattering ratios of 1.22±0.01 and 1.19±0.04, which are the same within errors.

The result for the sand size fraction is identical to the value calculated by Murray (1981) using thermoluminescence emissions from calcium fluorite, though in that study no uncertainty was quoted for the ratio.

The ratio for the fine silt sized fraction is statistically the same as previous estimates for fine grains using thermoluminescence (Doreen Stoneham, unpublished data).

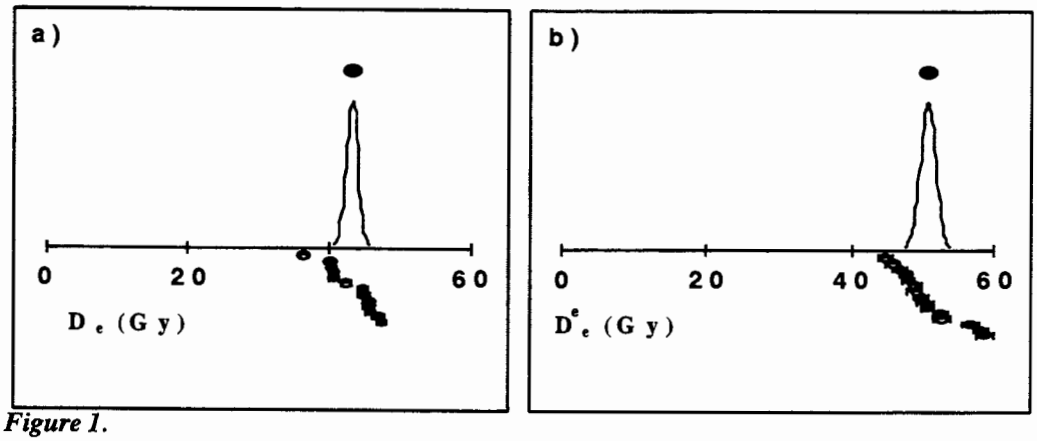

Figure 1.
Examples of SAR D_e populations for deep sea core samples 70KL 85-88
a) stainless steel substrate, b) aluminium substrate.

Aliquot	Substrate	
	Aluminium	Stainless Steel
1	8.78 ±0.01	7.26 ±0.03
2	8.78 ±0.02	7.22 ±0.04
3	8.80 ±0.01	7.37 ±0.02
4	9.21 ±0.03	7.38 ±0.02
Mean	8.89 ±0.11	7.31 ±0.04
Ratio	SS/Al	0.82 ±0.01

Table 1. *Equivalent Dose (Gy†) and summary ratio data for gamma irradiated annealed sand-sized quartz.*

Aliquot	Substrate	
	Aluminium	Stainless steel
1	58.8 ±1.3	44.8 ±0.7
2	66.4 ±1.7	46.9 ±0.8
3	46.7 ±1.1	47.2 ±0.7
4	52.6 ±1.2	36.5 ±0.6
5	50.3 ±1.2	48.6 ±1.0
6	52.5 ±1.0	40.0 ±0.5
7	48.9 ±1.1	44.5 ±0.7
8	50.5 ±0.8	45.5 ±0.7
9	48.0 ±0.7	42.2 ±0.7
10	49.8 ±1.0	40.5 ±0.6
11	45.8 ±0.8	45.6 ±0.7
12	47.7 ±0.8	40.4 ±0.5
13	44.7 ±0.9	40.2 ±0.5
14	56.7 ±1.1	45.4 ±0.7
15	48.8 ±0.8	
16	58.2 ±1.2	
Mean	51.7 ±1.4	43.5 ±0.9
Ratio	SS/Al	0.84 ±0.03

Table 2. *Equivalent Dose (Gy†) and summary ratio data for silt-sized quartz from deep sea sediments.*

Summary

Minimizing systematic uncertainties in optical and thermoluminescence dating is essential to make full use of the potential for high precision measurements now available to dating practitioners via single aliquot and single grain equivalent dose determination procedures. These new approaches can also usefully be exploited in reducing errors associated with absolute or relative alpha and beta source calibration, and laboratory irradiation related dose rate and backscattering conversion factors. This study has confirmed the previous backscattering efficiency ratios for coarse grains and confirmed the equivalence of the ratio for fine silt size dating fractions. It has additionally demonstrated that errors in such parameters can easily be reduced considerably (to better than 2-4%). Given that many other important ratios (e.g., coarse/fine dose rate) are likewise based on old techniques and technologies, it would be sensible for the community to systematically re-evaluate all such parameters, with known and low levels of uncertainty.

Acknowledgments

We are grateful to the review comments of Michel Lamothe and the patience of Didier Miallier.

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Reviewer
Michel Lamothe

Comments

The manuscript fully deserves to be published in Ancient TL. It is a nice and brief demonstration that the development of the SAR approach in luminescence opens a whole range of investigations. Some of this will hopefully concern the early work revolving around calibration procedures. This note might revive our need to initiate a true inter-calibration program between laboratories of radioactive sources, or to check for luminescence efficiency between the different types of irradiation and s.o.

Thesis abstracts

Thesis title: Application des méthodes de datation par luminescence optiquement stimulée à l'environnement océanique de l'Atlantique Nord
Translated title: Application of optically stimulated luminescence dating methods to the North Atlantic oceanic environment.

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In this study, the optically stimulated luminescence dating method was applied to Pleistocene sediments from three cores from the northern sector of the North Atlantic. The drill site Troll 91/8903 lies in the middle of the Norwegian Channel in the north-eastern region of the North Sea. The second core, SAB-85, is from Sable Island along the outer edge of the continental shelf of the coast of Nova Scotia. The third core (TWC/PC 84-030-001) comes from the abyssal plain of the southern sector of the Labrador Sea. The location of these three sediment cores provides a valuable record of Quaternary climatic fluctuations and ice sheet volumes in the circum-Atlantic zone. The analyzed samples represent marine and glacio-marine units, and their chronostratigraphic positions have been determined using lithologic, biostratigraphic and amino-stratigraphic criteria, and a limited number of radiocarbon dates.

In order to date these samples, potassium feldspar was used as the natural dosimeter and the sediments were dated using the Infrared Stimulated Luminescence (IRSL). Analyses were performed on polymineralic fine grains (4-11 μm), and K-feldspar coarse grains (125-250 μm) and the IRSL ages were calculated using multiple and single aliquots. The latter included both the single grain and the single aliquot regeneration (SAR) methods. In order to measure the equivalent dose (D_e), we applied the additive dose, regeneration, and Australian slide methods, the latter of which combines the first two.

The apparent IRSL ages obtained from the polymineralic 4-11 μm fraction from core Troll 8903 are generally in agreement with the assumed sediment ages based on the regional chronostratigraphy. Together, the good reproducibility of the results, and the observation of a plateau of equivalent dose values plotted as a function of stimulation time would have indicated that the IRSL signal of the samples was reset to zero at the time of sediment deposition. Nevertheless, this type of plateau is also observed for partially bleached diamictic sediments. Analysis of the coarse, monomineralic K-feldspar fraction revealed indeed a heterogeneous collection of well to partially bleached grains, this being based on the variability of the parameter R_I ($L_{N+\gamma}/L_N$). The R_I values measured after a delay of several days demonstrated that the IRSL signal was unstable and affected by fading. This phenomenon incurs an underestimation of IRSL ages. The *fadia* method, which uses the relationship between $R_{I(t1)}$ and $R_{I(t2)}$, allowed for an estimation of fading, which varied between 7 and 30 %. For some of the fine-grained samples (4-11μm), it appears that the agreement between apparent IRSL ages and assumed ages is therefore the result of the combined yet opposing effects of partial bleaching and fading.

The IRSL ages obtained for the samples from core SAB-85 (Nova Scotia) indicate that the major glaciation period that affected the Eastern Canada continental platform predates the Early Wisconsinan and probably prevailed until Late Illinoian (isotopic stage 6), or even earlier. Evidence of this major glaciation includes, among other facts, the incision from sub-glacial channels that became filled with the sediment sequence sampled by drill hole SAB-85.

A modified version of the single aliquot regeneration method revealed that both reproducibility and precision of the results were excellent. This technique was tested on fine-grained polymineralic and coarse-grained K-feldspar samples from all three cores. In contrast to the coarser grains aliquots, the estimated equivalent doses for the fine-grained fraction displayed considerable homogeneity between aliquots, suggesting that the sediment was reset to zero. These samples, however, are affected by fading on the order of 2.5 to 5 % per decade. IRSL ages corrected for fading are therefore greater than the presumed ages. Some of this overestimation is the result of thermal transfer within the natural aliquot upon the first preheating.

(Translated and edited from original French version)

Thesis title : Application des méthodes de datation par luminescence optique à l'évolution des environnements désertiques - Sahara occidental (Maroc) et Iles Canaries orientales (Espagne)
Translated title : Application of optically stimulated luminescence dating techniques to desert environment evolution - Western Sahara (Morocco) and Eastern Canary Islands (Spain)

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This paleoenvironmental study establishes a geochronological framework for the alternating arid and humid phases in Western Sahara and Eastern Canary Islands. This work is one component of a multidisciplinary study on global changes (Earth Processes in Global Change, EPGC) included within the scope of the CLIP project (Climate of the Past), sponsored by UNESCO (United Nations Educational, Scientific and Cultural Organization) and IUGS (International Union of Geological Sciences).

Optically stimulated luminescence (OSL) dating methods were applied to feldspar (IRSL) and, when possible, to quartz (GSL) from sediments that represent successive humid phases in three sections: Tah in Western Sahara, Rosa Negra and Mala on the islands of Fuerteventura and Lanzarote. Various methods inherent to the OSL technique (additive dose, regeneration, Australian slide, SAR, SAAD) were applied to multiple aliquots, single aliquots or single grains. A fading correction was applied to the feldspar ages. In the absence of independent chronological control, this comparative approach was considered necessary in order to validate the resultant ages.

In the Tah section (Western Sahara), different OSL methods gave comparable quartz and feldspar ages for every samples for which the OSL of quartz was far from saturation. It was found that the Tah section spans a period of 110 thousand years marked by humid phases at 107 ± 11 ka, 50 ± 5, 31 ± 3 and 13 ± 1 ka. The latter was dated using ^{14}C. The first event is thought to be a more pronounced humid phase.

In the Eastern Canary Islands, the application of OSL methods was restricted by mineralogical constraints caused by elevated carbonate contents and low concentrations of quartz and feldspar. In addition, the dated feldspar minerals show apparent sensitivity changes, probably due to their volcanic origin. Only the SAR method provided reliable results due to its advantage of properly correcting the luminescence sensitivity changes from single aliquots. Quartz was not dated since this mineral was absent from the topmost units and those grains present in the oldest units were saturated. Once the fading correction had been applied, the Rosa Negra section at Fuerteventura revealed the existence of humid phases at 253 ± 27, 190 ± 50 and 147 ± 25 ka. On Lanzarote, the basal samples from the Mala section could not be dated due to low IRSL emission and low reproducibility. Nevertheless, two humid phases in the upper horizons were dated at 191 ± 55 and 130 ± 11 ka. These ages correlate with those from the Rosa Negra section, suggesting that the *lacunae* observed at Rosa Negra carry regional significance.

A composite sequence was obtained by combining the Tah section with the apparently contemporaneous Rosa Negra and Mala sections. The newly compiled sequence reflects the dry to humid transitions that affected the Sahara during the past 250 000 years. These seems to coincide with interglacial or interstadial boundaries defined by stable isotope ($\delta^{18}O$) variations recorded in marine cores from the Canarian Basin, the latter being synchronised with the SPECMAP curve. In addition, the humid phases correspond to those recorded in travertine or high lake levels scattered throughout the rest of the Sahara and Sahel. With the exception of the horizon that should represent the 80 ka humid period (e.g. 5a isotopic stage) which was probably eroded by eolian deflation, these sections represent a unique land sequence covering several climatic cycles, found in an arid environment within the Saharian intertropical zone.

(Translated and edited from original French version)

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